

**THE USE OF CARBONATED CALCIUM-RICH MATERIALS AS
ADMIXTURES IN THE PRODUCTION OF OPC-BASED CONCRETE**

BY

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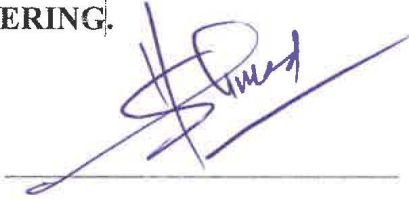
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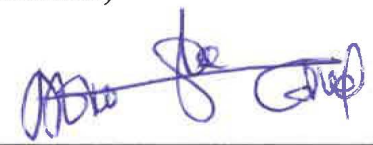
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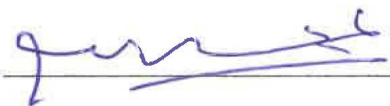


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I dedicate this work to my parents and wonderful siblings

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LIST OF ABBREVIATIONS

CRMs	:	Calcium-rich Materials
LSP	:	Limestone Powder
OPC	:	Ordinary Portland Cement
CC	:	Carbonated OPC
CL	:	Carbonated Lime
E-CO₂	:	Embodied Carbon Dioxide
EE	:	Embodied Energy

ABSTRACT

Full Name : [OMER ELZUBAIR OSMAN AHMED]

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Accelerated carbonation treatment is gaining significant attention as a viable means to reduce the carbon footprint in the concrete industry. However, the highly concentrated CO₂ environment required to carbonate concrete limits its application to pre-cast concrete. Recent studies attempted to address this limitation by incorporating CO₂ directly into the fresh concrete mixtures but their approaches remain challenging for on-site concrete mixing. To overcome this, the current study proposes introducing mineral carbonation into fresh concrete mixtures in three steps. Selecting hydrated lime and ordinary Portland cement (OPC) as calcium-rich materials (CRMs), this study proposes injecting CO₂ into slurries of these CRMs, powdering the carbonation products, and then incorporating them into concrete mixtures. Calcite was found as the dominant component in the carbonated CRMs, as evidenced by distinct calcite peaks observed in both thermogravimetric analysis (TGA) profiles, X-ray Diffraction (XRD) spectra, and morphology changes noted in scanning electron micrographs. The incorporation of carbonated CRMs in paste mixtures at OPC replacement levels of up to 15% sufficiently preserved the hydration products, as revealed by TGA and XRD. This resulted in enhanced early-age strength and sustaining above 95% of the compressive strength after 28 days. Compressive strength results revealed that at 10% replacement level, mortar mixtures with CC and CL show better performance than those with LSP, retaining 95% of the control's strength and elastic modulus, while LSP mixture maintained 87%. Tensile strength, drying shrinkage, and water absorption were better in CC and CL mixtures compared to LSP. CC mixtures exhibited the most insignificant rise in chloride migration coefficient even at maximum replacement level, exceeding control mixture's value by 28%. A 20% replacement level is identified as optimal for balancing environmental benefits with strength. This percentage was used to produce blended concrete mixtures, which demonstrated compressive strength, elastic modulus, and water absorption comparable to the control mixture, suggesting that the proposed carbonation method offers a path to sustainability in concrete production without significantly compromising its performance. Sustainability analysis of mortar/concrete mixtures suggested that incorporating carbonated CRMs in mortar/concrete can effectively reduce the embodied CO₂.

ملخص الرسالة

الاسم الكامل: عمر الزبير عثمان احمد

عنوان الرسالة: استخدام المواد المكرينة الغنية بالكالسيوم كمضافات في إنتاج الخرسانة

التخصص: الهندسة المدنية

تاريخ الدرجة العلمية: مايو 2024

تحتل معالجة الكربنة المعجلة باهتمام كبير كوسيلة فعالة لتقليل البصمة الكربونية في صناعة الخرسانة. ومع ذلك، فإن بيئة ثاني أكسيد الكربون عالية التركيز المطلوبة لكربونات الخرسانة تحد من تطبيقها على الخرسانة مسبقة الصب. حاولت الدراسات الحديثة معالجة هذا القيد من خلال دمج ثاني أكسيد الكربون مباشرة في الخلطات الخرسانية الطازجة، ولكن أساليبها تظل صعبة بالنسبة لخلط الخرسانة في الموقع. للتغلب على ذلك، تقترح الدراسة الحالية إدخال الكربنة المعدنية إلى الخلطات الخرسانية الطازجة في ثلاث خطوات. من خلال اختيار الجير المطفأ والأسمنت البورتلاندي العادي (OPC) كمادة غنية بالكالسيوم (CRMs)، تقترح هذه الدراسة حقن ثاني أكسيد الكربون في ملاط هذه الأسمنت البورتلاندي، وطحن منتجات الكربنة، ثم دمجها في الخلطات الخرسانية. تم العثور على الكالسيوم باعتباره العنصر المهيمن في CRMs المكرينة، كما يتضح من قمع الكالسيوم المتميزة التي لوحظت في كل من ملفات تحليل قياس الحرارة الحراري (TGA)، وأطياف حيود الأشعة السينية (XRD)، والتغيرات المورفولوجية التي لوحظت في مسح الصور المجهرية الإلكترونية. أدى دمج CRMs المكرينة في مخاليط المعجون بمستويات استبدال OPC التي تصل إلى 15% إلى الحفاظ بشكل كافٍ على منتجات الترطيب، كما كشفت TGA و XRD. أدى ذلك إلى تعزيز القوة في سن مبكرة والحفاظ على أكثر من 95% من قوة الضغط بعد 28 يومًا. أظهرت نتائج مقاومة الانضغاط أنه عند مستوى استبدال 10%، أظهرت خلطات الملاط ذات CC و CL أداء أفضل من تلك ذات LSP، حيث احتفظت بـ 95% من قوة التحكم ومعامل المرونة، بينما حافظت خلطة LSP على 87%. كانت قوة الشد والانكماش الجاف وامتصاص الماء أفضل في مخاليط CC و CL مقارنة بـ LSP. أظهرت مخاليط CC الارتفاع الأكثر أهمية في معامل انتقال الكلوريد حتى عند مستوى الاستبدال الأقصى، متجاوزة قيمة خليط التحكم بنسبة 28%. تم تحديد مستوى الاستبدال بنسبة 20% على أنه الأمثل لتحقيق التوازن بين الفوائد البيئية والقوة. تم استخدام هذه النسبة لإنتاج مخاليط خرسانية مخلوطة، والتي أظهرت قوة الضغط، ومعامل المرونة، وامتصاص الماء مقارنة بخليط التحكم، مما يشير إلى أن طريقة الكربنة المقترحة توفر طريقًا للاستدامة في إنتاج الخرسانة دون المساس بشكل كبير بأدائها. يشير تحليل استدامة مخاليط الملاط/الخرسانة إلى أن دمج CRMs المكرينة في الملاط/الخرسانة يمكن أن يقلل بشكل فعال من ثاني أكسيد الكربون المتجسد

CHAPTER 1

INTRODUCTION

One widespread issue that the world is currently facing is environmental degradation caused by releasing CO₂ into the atmosphere [1]. The growth of industrialization and urbanization across the globe has resulted in a marked escalation in carbon emissions, thereby fueling a surge in the worldwide atmospheric levels of anthropogenic greenhouse gases CO₂. The phenomenon has resulted in the escalation of global temperatures and alterations to the Earth's climate [2]. Carbon is released into the atmosphere through various means including human respiration, deforestation, oceanic processes, and the burning of fossil fuels. Sources of carbon emissions are divided into natural and artificial, and prior to the Industrial Revolution, natural sources of carbon emissions were generally disregarded. The Industrial Revolution resulted in a notable elevation of carbon dioxide levels as a result of anthropogenic emissions [3].

If the issue of CO₂ emission is not addressed, atmospheric CO₂ levels are expected to continue rising. According to reports in 2019, the mean concentration of CO₂ in the Earth's atmosphere was reported to be 414.7ppm, which was 45% higher than the levels recorded between 1980 and 1990 [4]. Additionally, data shows that the concentration of CO₂ has gone up by more than 2ppm annually in the past decade [5]. Furthermore, CO₂ emissions resulting from human activities have seen a rise of over 400% since 1950 [6].

According to recent research, there is a growing trend towards the capture, utilization, and storage of CO₂. Multiple approaches, including post-combustion, pre-combustion, and oxy-combustion, have been devised to capture CO₂ [7,8]. Using renewable energies along with CO₂ utilization is the most environmentally friendly approach to producing fuels, chemicals, and plastics. This method can significantly contribute to a sustainable economy and directly aligns with nine of the UN's 17 Sustainable Development Goals [9].

Worldwide, the cement manufacturing sector accounts for roughly 7% of all carbon dioxide emissions. The construction industry is the largest contributor to CO₂ emissions, having produced around 9.95 Gt/y of CO₂ in 2019 [10,11]. Despite of around 43% of the carbon dioxide emissions produced during cement production between 1930 and 2013 have been stored in cement materials through a process called natural carbonation [12]. However, this process occurs in a slow rate, thus, an accelerated carbonation process is needed to rapidly capture CO₂ [13].

Previous research has shown that the use of highly concentrated and pressurized CO₂ to carbonate concrete at an early stage can increase concrete strength [14,15]. This process involves placing early-stage concrete samples in a sealed chamber filled with CO₂ gas [16]. Once inside concrete, the CO₂ reacts with the fresh concrete to form calcium carbonates

(CaCO₃) [13]. However, the slow diffusion of CO₂ into the concrete limits the efficacy of this approach [17]. Furthermore, the process requires a high concentration of CO₂, making it applicable only to precast concrete. In an attempt to overcome the drawbacks of the previously mentioned technique, a fresh approach has been suggested by X. Qian et al. [18], involving the incorporation of CO₂ into concrete as an admixture. Their approach involves absorbing CO₂ using slaked lime as an absorbent, which then produces carbonates, and subsequently mixing it with other components to create concrete. They found that this method enhanced the hydration process and increased the volume of the hydration product of the OPC-based mortar specimens, and hence, compressive/flexural strengths were improved. The bottom line of their research suggests that calcium-rich supplementary cementitious materials (SCMs) can be utilized instead of slaked lime. This research intends to use variant alkaline materials as absorbents to characterize the product, obtain the optimum dosage of every absorbent and the duration of carbonation, and investigate its impact on the properties of the cement mixtures.

1.1 Need for Research

The need for research is underscored by the promising but limited successes of existing carbonation technologies in enhancing the properties of concrete. While initial results indicate improved strength and reduced shrinkage with the addition of low percentage of carbonated lime, comprehensive studies are needed to investigate higher replacement levels. Additionally, practical challenges related to the on-site application of carbonation techniques highlight the necessity for innovative approaches that simplify the process. To address these identified research needs, this study proposes the development of dry powdered forms of carbonated calcium-rich materials (OPC and lime), which could be easily stored and used on-site, offering a viable solution to bypass current logistical hurdles and potentially expand CO₂ utilization in construction.

1.2 Research Objectives

The main objective of this study is to investigate the influence of a new approach of carbonating different calcium-rich materials and using them as admixtures for producing OPC-based mixtures with enhanced properties. The sub-objectives are as follows:

1. To obtain the optimum dosage and carbonation duration for each of the selected calcium-rich materials (OPC, and lime).
2. To analyze the microstructure of carbonated slurries.
3. To analyze the microstructure of the produced paste.
4. To evaluate the impact of using carbonated calcium-rich materials on the mechanical and durability properties of mortar/concrete.
5. To evaluate the beneficial impact on the environment.

CHAPTER 2

LITERATURE REVIEW

2.1 Background

Global warming and climatic changes are driven by the rise in CO₂ emissions from the growth of industrialization and urbanization [1,2]. Carbon is released into the atmosphere through various means, including human respiration, deforestation, oceanic processes, and the burning of fossil fuels. While carbon emissions stem from both natural and anthropogenic sources, it is the surge in CO₂ levels attributed to human activities since the industrial revolution that has markedly amplified the phenomenon [3].

In 2019, the average concentration of CO₂ in the atmosphere was reported to be 414.7 ppm, which was 45% higher than the levels recorded between 1980 and 1990 [4]. Additionally, data shows that the concentration of CO₂ has gone up by more than 2 ppm annually in the past decade [5]. Furthermore, CO₂ emissions resulting from human activities have seen a rise of over 400% since 1950 [6]. The levels of CO₂ in the atmosphere are expected to keep rising if the issue of CO₂ emission is not tackled. However, recent reports indicate a growing trend towards the capture, storage, and utilization of CO₂ [7,19–24].

Mineral sequestration is a carbon sequestration technique that assumes a critical function in regions where geological sequestration is impractical due to concerns such as subterranean leakage or insufficient subterranean cavities [25–28]. It is considered the safest approach to achieve maximum CO₂ emission sequestration capacity Table 1. The fundamental concept of mineral sequestration involves the natural process of weathering, where CO₂ undergoes a reaction with minerals containing magnesium or calcium to yield carbonates that are both stable and ecologically benign [29,30]. Magnesium and Calcium are abundantly present in the Earth's crust, accounting for approximately 2.0% and 2.1%, respectively, making minerals containing these elements ideal for the process of CO₂ sequestration [31].

Table 1: Comparison of various carbon dioxide routes

Storage type	Advantages	Disadvantages	Capacity (Gt)	Ref.
Ocean	Plenitude. Less monitoring.	High environmental hazard.	1000	[32]
Geological	Acceptable environmental impact. Economical.	Leakage threat. Constant monitoring.	1800	[33]
Biological	Inexpensive. Low environmental impact.	Inadequate capacity. Potential for food chain disruption.	100	[34]
Mineral	Environmentally friendly. Long-lasting. Effective use of industrial byproducts.	Expensive. Requires significant energy. Extraction of raw materials results in environmental concerns.	10000 to 100000000	[35]

The mineral sequestration process, which involves capturing and storing carbon dioxide (CO₂) using alkaline minerals, can be carried out in two ways: in-situ, which occurs below the ground level, and ex-situ, which occurs above ground level [25,36–39]. In the in-situ sequestration process, CO₂ is injected into underground reservoirs, where it reacts with alkaline minerals in geological formations to produce carbonates [40]. Conversely, in case of the ex-situ sequestration, preliminary procedures such as rock excavation and mineral ore comminution are executed for preparing the minerals for their CO₂ sequestration [41]. Additionally, incorporating industrial waste into ex-situ sequestration not only leverages the recycling of waste materials but also enhances the sustainability and efficiency of the sequestration process[42].

2.2 CO₂ capture and sequestration in concrete

During the process of cement production, three primary sources are responsible for generating carbon dioxide. The first source is the calcination process entailing the breakdown of CaCO₃ into CaO and CO₂, which accounts for about 50% of the total CO₂ emissions. The second source is the combustion of fuels in the kiln, which generates approximately 40% of CO₂ emissions. The remaining 10% of emissions arise from ancillary manufacturing activities, including the transportation and electricity consumption [43–46].

Carbonation, a process involving the binding of CO₂ with other materials, occurs naturally and may contribute to mitigating climate change. However, accelerated carbonation is a more feasible and efficient solution for the cement industry. Accordingly, various carbonation routes have been developed that offer viable solutions to mitigate the effects of these emissions on a short-term basis (Figure 1, Table 1). The carbonated materials obtained from the mineral sequestration of CO₂ in carbonatable materials can be used as substitutes for conventional ingredients in concrete production. Other accelerated carbonation routes (accelerated carbonation curing and mixing) can also be used to capture CO₂ emitted during cement production and convert it into stable forms of calcium carbonate, thus reducing the carbon footprint of the cement industry.

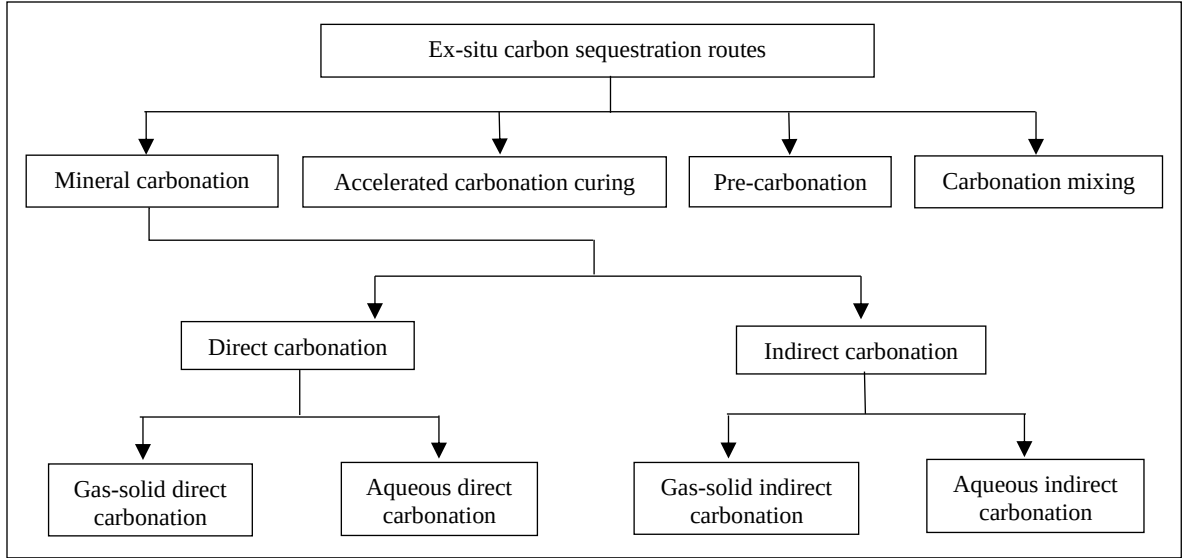
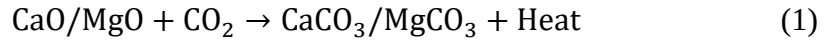


Figure 1: Classification of ex-situ carbon sequestration routes

2.2.1 Mineral Carbonation

Mineral carbonation involves fixing (sequestering) CO₂ in stable carbonate forms, such as CaCO₃ and MgCO₃, into carbonatable materials. In early mineralization studies, mineral CO₂ fixing was achieved by accelerating the natural process of carbonating silicate rocks. The result is permanent storage of CO₂ [13] in these materials. This process is represented by the general mineralization reaction presented in Eq.(1).



In later mineralization studies, CO₂ sequestration was also carried out using alkaline solid waste materials, such as steel slag, cement waste, and coal fly ash, instead of natural resources. This saves the nature and reduces energy consumption in the grinding and extraction of carbonatable earth materials [47]. Alkaline solid waste materials are well-suited for mineral carbonation due to their high reaction rates, low energy requirements, and superior carbonate conversion efficiency when compared to natural minerals, in which the carbonation occurs at a slow rate. Additionally, the mineral carbonation of solid waste not only provides a means of waste management but also offers the added benefit of reducing CO₂ emissions [47–49]. Mineral carbonation is divided into two main categories, namely direct and indirect carbonation.

Direct mineral CO₂ sequestration

Liu et al. [50] describe the direct CO₂ sequestration as the simplest carbonation approach, which involves only one step for carbonation. This technique can be implemented in two ways: gas-solid carbonation and direct aqueous carbonation methods. When comparing

different carbonation methods, direct aqueous carbonation is frequently used as a benchmark [41].

1. Gas-solid carbonation

Gas-solid carbonation is the simplest reaction that occurs when a solid containing Ca or Mg reacts with CO₂ gas at proper temperature and pressure [51]. Direct gas-solid carbonation has numerous benefits, including a high capacity and a rapid rate of CO₂ adsorption. As a result, it holds great potential for capturing and storing carbon dioxide for in-situ applications [51]. However, despite the use of high temperatures and pressure, the reaction rate of this method is often inadequate and slow [11,52]. Table 2 shows gas-solid carbonation treatment carried out on numerous solid wastes.

Table 2: Gas-solid CO₂ sequestration of different solid wastes

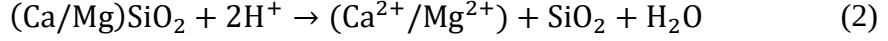
Solid waste	Carbonation condition	CO ₂ uptake	Carbonation efficiency	Remarks*	Ref.
Steel slag	(100% CO ₂ , atmospheric, 600°C) for 1 h.	8.85%	55.5%	39.68% CaO	[53]
Linz–Donawitz/converter (LD) steel slag	(20% CO ₂ , 80% Ar, 0.2 bar, 90°C) for 48 h.	1.5%	35%	Average particle size of 2-3 mm. 5.4% CaO	[54]
Electric arc furnace (EAF) slag	100% CO ₂ , 3 bar, 30°C) for 3 h.	1.1%	3.7%	28.27% CaO	[55]
Fly ash (FA)	(100% CO ₂ , 1-15 bar, 25/45°C) for 2 h	18.2%	74%	31.9% CaO	[56]
FA	(100% CO ₂ , 10 bar, room temperature) for 1 h.	2.63%	35.5%	28.42% CaO	[57]
FA	(100% CO ₂ , 20 mL/min, 160°C) for 4 h.	11.7%	48%	35% CaO	[58]
Recycled concrete aggregate (RCA)	(100% CO ₂ , atmospheric, 25°C) for 24 h.	3.8%	-	Particle size between 5-10 mm	[59]
RCA	(100% CO ₂ , 0.1 bar, 40°C) for 24 h.	5%	-	Particle size between 1-4 mm Porosity ≈ 13.8 %	[60]
RCA	(100% CO ₂ , 2.7 bar, 21°C) for 24 h.	1%	-	Particle size between 12-19 mm	[61]

*The CaO values are the total CaO quantities obtained from chemical analysis.

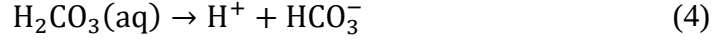
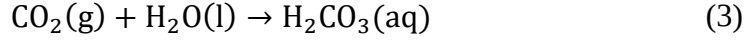
2. Aqueous carbonation

The gas-solid reaction turns into a gas-liquid-solid reaction when water is added, which boosts the carbonation reaction rate. The process of direct aqueous carbonation can be described in three distinct stages (as illustrated in Figure 2) [62–64]:

a. Initial leaching of minerals



b. Dissolution of CO_2 resulting in the release of carbonate ions



c. Subsequent nucleation and growth of mineral carbonates

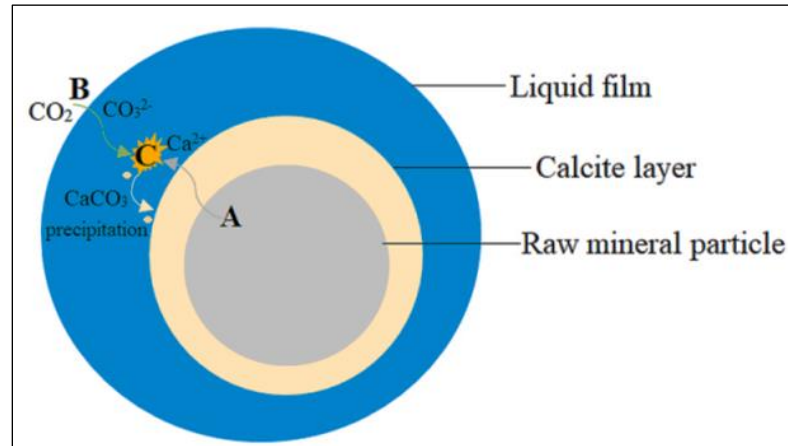
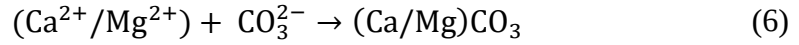


Figure 2: Schematic diagram of aqueous carbonation [65]

These three steps take place in a single reactor [66–68] where CO_2 is dissolving into water to create carbonic acid, which then breaks down into H^+ and CO_3^{2-} (equation (3)-(5)) [41]. The acidic solution resulting from H^+ causes the minerals' Ca/Mg in the matrix to dissolve and react with CO_2 to create calcium/magnesium carbonate equation ((2), (6)) [41]. Table 3 shows aqueous carbonation treatment carried out on numerous solid wastes.

Table 3: Aqueous carbonation of different solid wastes.

Solid waste	Carbonation condition	CO_2 uptake	Carbonation efficiency	Remarks	Ref.
Basic oxygen furnace (BOF) slag	(100% CO_2 , atmospheric, 600°C) for 1 h.	46.5%	72%	31.05% CaO	[69]
Ground granulated blast furnace (GGBF) slag	(30 bar, 40°C, 60 rpm) for 48 h.	2.95%	8.83%	42.5% CaO	[70]

EAF slag	100% CO ₂ , 6 bar, 30°C) for 3 h.	8.2%	26.97%	28.27% CaO	[55]
FA	(100% CO ₂ , 40 bar, 25° C)	2.6%	82%	5% CaO	[71]
FA	(100% CO ₂ , 4 L/min, 10 bar, room temperature) for 1 h	5.03%	67.9%	28.42% CaO	[57]
FA	(100% CO ₂ , 40 bar, 90°C) for 2 h.	7.18%	86.4%	9.2% CaO	[72]
RCA	(100% CO ₂ , atmospheric, 25° C) for 10 min.	11.6%	-	Particle size between 5-10 mm	[73]
RCA	(98% CO ₂ , atmospheric) for 10 min.	10.7%	-	Particle size between 0.14-4 mm	[74]
RCA	(100% CO ₂ , 10 bar, room temperature) for 10 min.	5.7%	-	≈31% CaO. Particle size < 0.5 mm	[75]
Recycled concrete fines	(14% CO ₂ , 4 L/min, room temperature) for 1.5 h.	12%	-	-	[73]

Indirect CO₂ sequestration

Indirect carbonation, as opposed to direct carbonation, involves multiple stages (leaching and carbonation) [76–78]. The leaching stage involves the extraction of reactive elements (Mg/Ca) from minerals using acid or other solvents. In the carbonation stage, CO₂ reacts with Ca/Mg under alkaline conditions [52,79]. Unlike direct method, the effectiveness of indirect CO₂ sequestration is dependent on various parameters, such as temperature, pressure, and pH [80].

Although indirect carbonation has not been applied to carbonate cementitious materials, its detailed examination within this review serves as a foundation for understanding the broader implications of this technology in concrete. There are various approaches to indirect carbonation, which can be outlined as follows:

1. Indirect gas–solid CO₂ sequestration

In contrast to the direct carbonation process from silicate forms of magnesium or calcium, the carbonation process of magnesium or calcium oxides or hydroxides exhibits a considerably higher rate of reaction. However, it is rare to find Ca/Mg oxides/hydroxides in nature, and thus, they need to be produced from Ca or Mg silicates. As a result, there are several stages to the indirect carbonation method. Initially, Ca or Mg that has been extracted undergoes a conversion process into oxides/hydroxides. After their formation, these hydroxides or oxides of minerals undergo carbonation under arid conditions. The utilization of steam has the potential to augment the reactivity of calcium and magnesium hydroxide (Ca/Mg(OH)₂) [81].

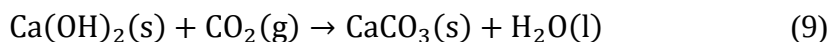
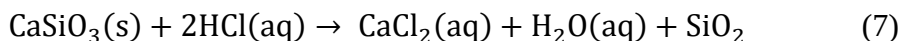
According to Lackner et al. [82], it was observed that at 500 °C and 340 bar, it takes approximately 2 hours for complete conversion of Mg(OH)₂ to MgCO₃. On the other hand, Butt et al. [83] were able to attain a conversion rate of approximately 90% for the transformation Mg(OH)₂ into MgCO₃ in just 30 minutes at a temperature of 565 °C and a

pressure of 53 bar with a particle size of 0.1 μm . Despite the rapid carbonation rates associated with this method, its primary disadvantage lies in its high energy demands, particularly for the endothermic step of extraction. Therefore, additional investigation is needed to optimize hydroxides extraction.

2. Indirect aqueous CO₂ sequestration

This approach utilizes various chemicals, such as H₂SO₄, HCl, and NH₄Cl to enhance the process of extracting metal ions [38,84] to make them available for reaction with CO₂. The following subsections explain the main indirect aqueous carbonation methods employed in previous studies.

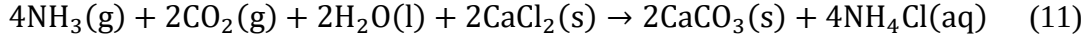
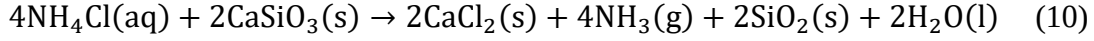
Hydrochloric acid: This approach was proposed by Lackner et al. in 1995 [85], where calcium carbonates are produced from calcium silicates through a three-stage process using hydrochloric acid in an indirect aqueous carbonation approach. In the first step, minerals are extracted with the help of 1 M HCl (equation (7)). Next, the solution is heated to a temperature range of 100 – 250°C, which corresponds to the vaporizing point of water, resulting in the precipitation of Ca(OH)₂ and enabling the concurrent recovery of HCl (equation (8)). Subsequently, the resultant Ca(OH)₂ is dissolved in an aqueous medium and reacts with CO₂ to generate CaCO₃ (equation (9)). It's important to note that the reaction step presented in equation (8) is thermodynamically unfavorable, thus further investigation is needed to elucidate the chemical process leading to the reported HCl recovery.



One of the primary drawbacks associated with this approach pertains to the expenses incurred in utilizing hydrochloric acid, owing to the energy-intensive process of evaporating the aqueous solution. This can result in a cost of over \$150 per ton of CO₂ [86]. Thus, the method is not feasible for capturing carbon dioxide from alkaline solid waste minerals [87][88].

pH swing procedure is a sequential process utilized in aqueous carbonation, comprising of two primary stages. The initial stage involves a reduction in the pH of the solution to facilitate the extraction of mineral ions. Subsequently, the pH is elevated in the subsequent stage to encourage the precipitation of carbonates [78,89,90]. Ammonium salts are used in this process to improve the efficiency of mineral dissolution, as well as facilitate additive recovery and reuse. The procedure entails the chemical reaction between CO₂, calcium chloride (CaCl₂) and ammonia (NH₃), resulting in the formation of ammonium carbonate

$[(\text{NH}_4)_2\text{CO}_3]$ and a CaCl_2 solution. Subsequently, the solution obtained is conveyed to a precipitator, where the precipitation of CaCO_3 takes place under conditions of reduced pH. At this stage, the recovery of ammonium chloride (NH_4Cl) is accomplished (equation (11)). The recovered NH_4Cl is subjected to a reaction with CaSiO_3 in a separate vessel under high pH conditions, resulting in the regeneration of NH_3 and CaCl_2 for reuse (equation (10)) [91,92].



In summary, mineral carbonation processes arise as viable strategies for CO_2 sequestration and offer a dual advantage of addressing climate change and utilizing industrial wastes. The effectiveness of these carbonation processes, direct or indirect, ultimately depends on the precise quantification of CO_2 uptake capacity and carbonation efficiency. These parameters are crucial, as they not only assess the feasibility of the carbonation methods but also their contribution towards achieving significant reductions in CO_2 emissions.

Quantification of CO_2 uptake capacity and carbonation efficiency

The primary goals of employing accelerated carbonation process within the concrete sector encompass two key purposes, namely, reducing net carbon emissions and enhancing the efficiency of construction materials. These goals are intricately connected to a crucial element, namely, the ability of the materials to capture carbon dioxide. The CO_2 sequestration capacity denotes the percentage of net CO_2 absorbed in relation to the raw material before carbonation [69,93,94]. Equation(12) provides the mathematical representation of this concept. The determination of carbon dioxide absorption capacity can be achieved through a range of methodologies, including the mass curve method, the mass gain approach, and thermogravimetric analysis [95–97].

$$\text{CO}_{2\text{-uptake}} = \frac{M_{\text{CO}_2}}{M_c} * 100 \quad (12)$$

Where:

M_{CO_2} is the mass of CO_2 sequestered after the carbonation treatment.

M_c is the original mass of the material prior to carbonation treatment.

In addition to CO_2 sequestration capacity, carbonation efficiency, which is also referred to as the degree of carbonation, is a significant parameter for evaluating the carbonation impact of materials [98,99]. Equation(13) provides the formula for calculating this

parameter. It represents the amount of CO₂ successfully captured (M_{CO_2}) by a given material under particular conditions, in relation to the hypothetical CO₂ sequestration potential (M_{th}) calculated using Huntzinger's formula in equation (14) [100] or Steinour's formula in equation (15) [101]. In general, materials with higher carbonation capacities exhibit higher carbonation efficiencies.

$$CO_{2-eff} = \frac{M_{CO_2}}{M_{th}} * 100 \quad (13)$$

$$M_{th} (\%) = 0.785(CaO - 0.56CaCO_3 - 0.7SO_3) + 1.091MgO + 0.71Na_2O + 0.468(K_2O - 0.632KCl) \quad (14)$$

$$M_{th} (\%) = 0.785(CaO - 0.7SO_3) + 1.091MgO + 1.42Na_2O + 0.935K_2O \quad (15)$$

It should be emphasized that equations (14) and (15) serve as theoretical tools for estimating the potential CO₂ uptake capacity of materials under idealized conditions. They are particularly useful for evaluating the theoretical efficiency of materials in capturing CO₂, not for predicting the actual CO₂ uptake, which is influenced by numerous variables and should be measured experimentally.

2.2.2 Potential uses of carbonated waste materials

Aggregates substitutes

Carbonated recycled concrete aggregates (RCA) are promising substitutes for the conventional aggregates offering sustainable concrete production. It is reported that the cement-based concrete made with carbonated RCA has lower chloride penetration and drying/autogenous shrinkage than that produced using uncarbonated RCA [74,102–104]. Additionally, compressive and tensile strength of concrete incorporating RCA were evaluated in several studies [73,105–107]. It was found that substituting untreated RCA with carbonated RCA enhanced the strength of cement-based composite [108–110], however, the enhanced strength was significantly lower than that of the control using natural aggregates [111,112]. The enhanced strength due to carbonated RCA can be attributed to two main mechanisms [73,105,106,108,113]: (i) carbonation enhances the physical properties of RCA, resulting in lower porosity and increased strength, and (ii) improved bond between mortar and carbonated RCA. More specifically, the latter mechanism occurs when the CaCO₃ and silica gel present on the carbonated RCA surface interact with the newly formed hydration products of the mortar, resulting in the generation of a greater quantity of calcium silicate hydrates (C-S-H) gel [108,114].

Portland cement additives

The carbonated materials obtained from the treatment of industrial waste have shown potential as substitutes for Portland cement in concrete production. Although the research in this field remains limited, the preliminary results from the current investigations have demonstrated a favorable and encouraging outlook. Table 4 shows some results of studies conducted on using carbonated solid waste materials as supplementary materials in mortar.

Table 4: Utilization of carbonated solid wastes in concrete

Solid waste	Substitution percentage	CO ₂ uptake	Increase in 28-d strength	Ref.
Steel slag (SS)	10%	12%	23% (of untreated-SS samples)	[115]
Electric arc furnace reducing slag (EAFRS)	10%	38%	Comparable	[116]
FA	10%	3.2%	Comparable	[99]
High calcium fly ash (HFA)	10-30%	3.8-3.88%	5-12.5% (of untreated HFA samples)	[117]
Petroleum coke fly ash (PCFA)	5%	8.45%	21.7%	[118]
FA	25%	-	1.36% and 5.8% strength gain at 90 and 120 days of sulfate exposure. Robust resistance to salt, acid, and sulfate attack.	[119]
Concrete slurry waste (CSW)	20%	20.4%	8% (of untreated-CSW samples).	[120]

Generally, the strength enhancement observed in cement pastes and mortars due to the incorporation of carbonated waste materials can be attributed to two main factors. Firstly, during the carbonation treatment, the formation of small calcite particles occurs [121], which effectively fills the pores in mortars and leads to a reduction in porosity [115,118]. Secondly, the resulting calcite reacts with aluminates (C_3A), which aids in the creation of phases of calcium carboaluminate [116,122,123]. These mechanisms collectively contribute to the overall improvement in strength. Essentially, the incorporation of carbonated industrial wastes in concrete offers significant benefits such as increased volume stability, enhanced mechanical and enhanced durability properties, in contrast to their non-carbonated counterparts [65].

2.2.3 Accelerated carbonation curing (ACC)

ACC of concrete is a process that can occur during both the fresh and hardened states of the material. This process is known to result in increased strength and densification of the pore structure, as well as a reduction in chemical attack due to decreased permeability [109,124,125]. However, there is a potential concern regarding reinforcement corrosion, as the decrease in alkalinity that occurs during carbonation may contribute to this issue [126].

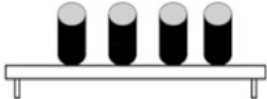
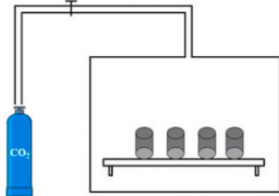
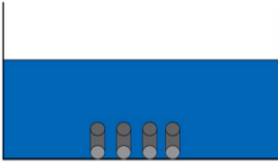
The process of ACC

ACC typically involves a three-stage methodology including pre-carbonation, carbonation, and post-carbonation, as detailed in Table 5.

I. Pre-carbonation

Following the casting of samples, this phase is initiated to optimize the conditions for carbonation. This stage involves in-mold curing, which aims to promote the initial setting of the cementitious composite. Once hardened, the specimens are removed from the molds to increase their surface area, thus enhancing water evaporation. The evaporation of free water is vital to improve CO₂ diffusion during carbonation. Nevertheless, it's important to avoid excessive water loss, which can hinder the carbonation reaction by causing dehydration [96,127]. Typically, pre-carbonation process takes place in conditions with temperatures of 20–25°C and relative humidity between 40–60% [128].

Table 5: ACC stages [128]

Stages	Stage 1: Pre-carbonation	Stage 2: Carbonation	Stage 3: Post-carbonation
Sketch			
Controlled factor	Temp (20–25°C) RH (40–60 %)	Temp (20–40°C). RH (>40 %). CO ₂ concentration (>10 %). CO ₂ pressure (1–5 bar). Duration (2–24 h).	Duration
Reaction	Cement hydration.	carbonation of unhydrated cement and hydration products.	Continued hydration of unhydrated cement.

II. Carbonation

Carbonation curing involves subjecting mixtures to a CO₂-rich environment over a certain period. This method is categorized into two reaction systems: enclosed and flow-through chambers. The flow-through system is recognized for its lower energy consumption, yet the enclosed system is favored for its superior reaction effectiveness, attributed to its ability to maintain a pressurized gas environment. Enclosed chambers are often preferred for laboratory-scale experiments, as they enhance CO₂ diffusion through the application of high-pressure gas, resulting in more efficient carbonation rates and extents [129,130].

Most research on the early-stage carbonation of OPC-based materials typically takes place at ambient temperatures (20–25 °C). However, the reaction rate can be significantly influenced by temperature variations. Higher temperatures can enhance CO₂ diffusion yet reduce its solubility in water and also lead to quicker evaporation of water. The leaching and migration of reactive ions might be impacted as well [131]. It's important to recognize that early-stage carbonation generate heat, with observed temperature rises of up to

approximately 58 °C in concrete and around 70 °C in cement paste under ambient conditions [16,132]. Introducing external heat to elevate temperature could intensify the peak temperature experienced during carbonation, potentially affecting the stability of reaction products and, consequently, the performance of the cured concrete.

For effective CO₂ diffusion and carbonation, a relative humidity within the ambient range is generally preferred. Carbonation curing studies on OPC often utilize a RH of about 60-70%. Higher humidity levels, exceeding 90%, are sometimes applied to minimize water evaporation under conditions of increased temperature [133] or when w/b ratio in the mixture is low [134].

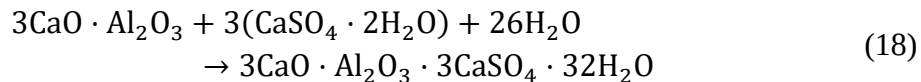
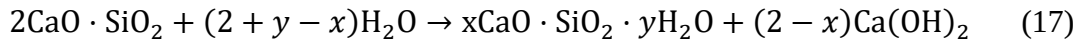
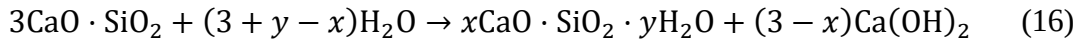
3. Post-carbonation

Following carbonation, post- carbonation is necessary for the further hydration of remaining unreacted cement particles. It's essential to compensate for water lost during pre-carbonation and carbonation at this phase to ensure the continuation of the hydraulic reaction. Initial findings have underscored the significance of curing post carbonation in contributing to extra increases in strength. It has been observed that hydrating for three days after carbonation can result in a strength enhancement of up to 45% [14]. Further research has underscored the importance of this subsequent hydration phase for optimizing strength outcomes [135] and improving pore structure [136]. Additionally, it has been noted that the formation of CaCO₃ during early carbonation can influence the hydration process, particularly by interacting with components like C₃A [136].

Chemical reactions during ACC

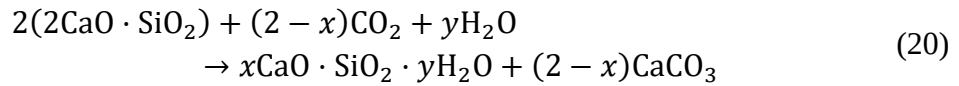
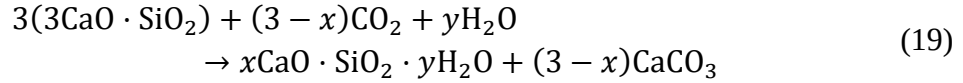
I. Carbonation of cement

Upon contact with water, cement undergoes hydration, with the primary reactions detailed in equations (16)-(18) occurring during pre-carbonation stage. The hydration level of cement paste can achieve 20-30% within the first 24 hours, aligning with the typical pre-carbonation duration [129,137].



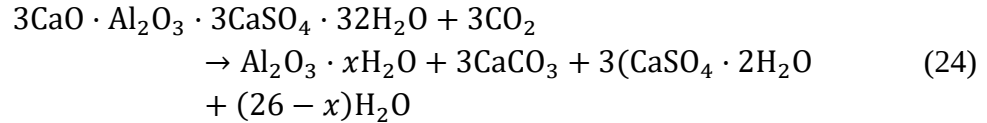
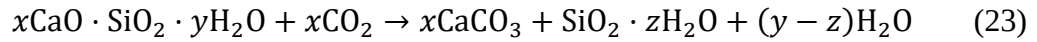
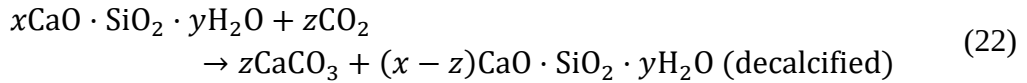
During carbonation stage, the reaction outlined in Figure 3 involves: a) CO₂ transport and dissolution, leading to carbonate ion formation; b) the discharge of Ca²⁺ ions into the pore solution from cement and its hydration products; c) the formation of calcium carbonate through the interaction of Ca²⁺ ions with carbonate ions [39,40]. The crystal development

of CaCO_3 is presented in Figure 3. The primary substances reacting with CO_2 are the unhydrated calcium silicates, C_3S and C_2S , as shown in the carbonation reactions for C_2S and C_3S (equation (19) and (20)). These reactions, occurring under moist conditions, result in the production of calcium silicate hydrate (C-S-H) and calcium carbonate. C_3A exhibits minimal reaction with CO_2 , showing a preference for hydration, although ettringite derived from hydrated C_3A may undergo carbonation [138]. Meanwhile, C_4AF has a significantly limited reaction with CO_2 , leading to the formation of calcite and minor amounts of vaterite, aragonite, and gibbsite [139].



II. Carbonation of hydration products

Hydration products, such as C-S-H and CH, are capable of undergoing reactions with CO_2 (equation (21)–(24)) [140]. Such carbonation processes initiate the decalcification of C-S-H, leading to its transformation into silica gel with a greater density than C-S-H. Additionally, ettringite can undergo reactions with CO_2 , resulting in its decomposition into various compounds [141,142]. Monosulphate also reacts with CO_2 , though its presence is minimal in the early stages of carbonation [97].



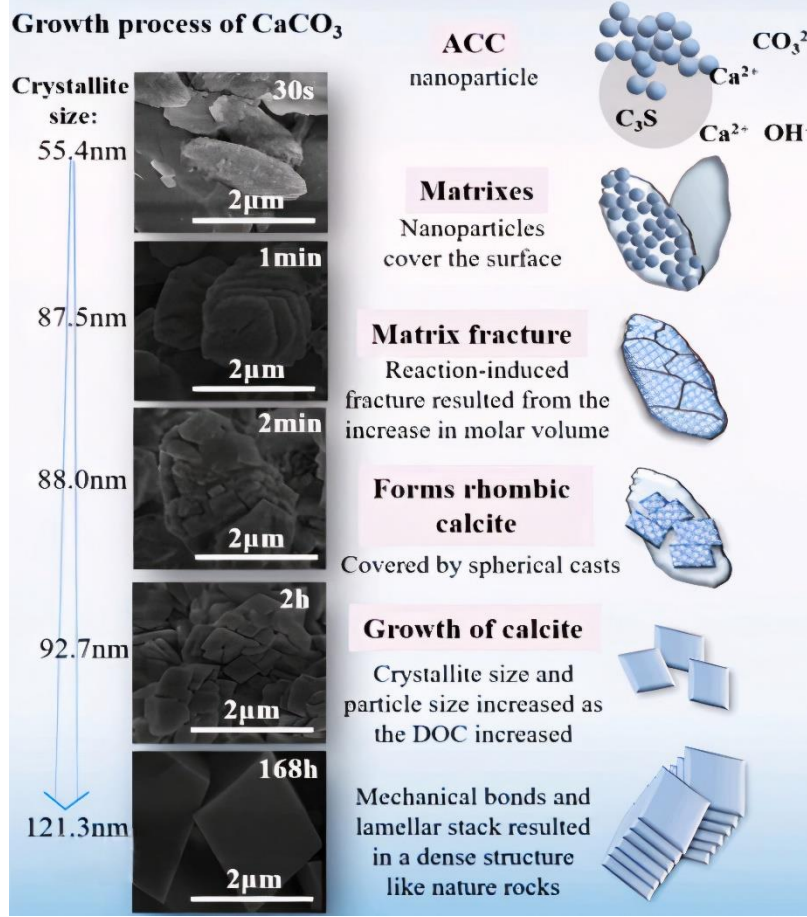
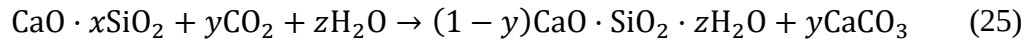


Figure 3: Schematic illustration of the crystal development process of CaCO₃ (reused with permission from [143])

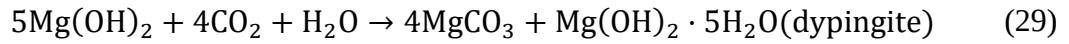
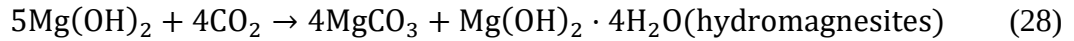
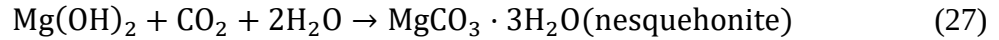
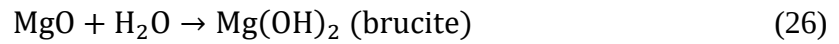
III. Carbonation of other binders

Materials that react with CO₂, like non-hydraulic calcium silicates (such as wollastonite) and mineral oxides (for example, MgO), can be employed as substitutes for a portion of cement in carbonation curing. This substitution helps in reducing the amount of cement used, thereby decreasing CO₂ emissions. Furthermore, the CO₂ sequestration capability of these materials can be significantly improved, given their substantial CO₂ absorption potential. Due to the complex composition of these materials compared to cement, their chemical behavior under carbonation curing is more complicated. Non-hydraulic calcium silicates like wollastonite [144,145], along with materials such as rich-calcium waste steel slag [146] and cement kiln dust [147], demonstrate significant reactivity towards CO₂. These minerals, reacting at varying rates with CO₂, can enhance the strength of cement-based composites through carbonation curing, as shown in equation (25).



Portlandite may also serve as a binder in cementitious composites following carbonation curing, thanks to its remarkable reactivity with CO₂, as indicated in equation (21) [148,149].

The potential of MgO as an alternative binding agent for carbonation curing in cementitious systems has garnered interest in recent studies [2,150–153]. MgO stands out for its excellent CO₂ capture capacity and requires a lower calcination temperature (700–1000 °C for MgCO₃ compared to 1450 °C for CaCO₃) [154]. The carbonation reactions of MgO, leading to the formation of nesquehonite, hydromagnesites, and dypingite, are shown in equations (26)-(29), which contribute to the binding characteristics in carbonated cementitious composites [155].



Supplementary cementitious materials (SCMs), such as steel slags and coal fly ash, have shown carbonation reactivity, which varies with the presence of reactive Ca/Mg-bearing phases [156,157], with their chemical interactions also represented by equations (25)-(29). The substitution of cement with SCMs or fillers can slow down hydration, potentially increasing early-age CO₂ diffusivity due to heightened permeability [158].

Moreover, as illustrated in Figure 4, the dispersion of cement particles is enhanced by the addition of CO₂-inert SCMs, improving their availability for CO₂ reaction. SCMs can act as nucleation sites, aiding in the formation of carbonation products [159]. Similarly, the use of non-reactive fillers, such as LSP, as a partial Portland cement substitute can significantly boost the efficiency of carbonation [160].

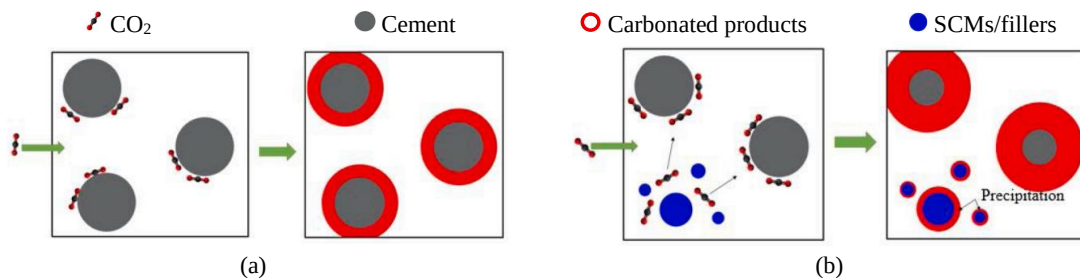


Figure 4: Influence of SCMs/fillers on carbonation characteristics: (a) absence of SCMs/fillers and (b) presence of SCMs/fillers (reused with permission from [161]).

Effect of ACC on the properties of cement-based concrete

Numerous studies [154,159,170–179,162–169] have extensively investigated the impact of ACC on various properties of concrete. Table 6 presents a concise summary of the effects of ACC on distinct properties of blended cement concrete.

Table 6: Effects of ACC on the properties of blended cement concrete

Property	Blended concrete type	Substitution percentage	Effect of ACC on the property compared to normal curing	Ref.
Compressive strength	Silica fume	10%	Increased.	[162]
	PCC ash	10%	Increased.	[163]
	Fly ash	20%	Increased.	[159]
	GGBF slag			
	CaO+MgO	5 – 15%	Increased.	[164]
	Steel slag+CaO+MaO	60% slag 20% CaO 10% MgO	Increased by 500%.	[165]
	Volcanic ash	20-50%	Increased.	[166]
Tensile strength	Fly ash	25%	Increased by 21%.	[167]
	Reactive MgO + Fly ash	-	Increased by 200%.	[154]
Drying shrinkage	LSP	20%	Increased but within the permissible limit.	[168]
	AOD slag	0-60%	Decreased as AOD slag replacement level increased.	[169]
	coal gangue	0-50%	Decreased as coal gangue replacement level increased.	[170]
	Slag	25-50%	Increased.	[171]
Modulus of elasticity	Fly ash	25%	Comparable.	[167]
Water absorption	Belite	5-15%	Reduced.	[172]
	Steel slag	-	Reduced.	[165]
Water penetration depth	OPC	-	Reduced.	[167]
	Fly ash	25%		
Chloride permeability	OPC	-	Reduced.	[167]
	Fly ash	25% ¹		
	Reactive MgO	50% ²	Reduced.	[173]
Chloride diffusion	Biochar	2-5% ²	Higher biochar ratios led to lower chloride diffusion coefficients.	[174]
	AOD slag	0-60% ³	Higher AOD slag ratios led to lower chloride diffusion coefficients.	[169]
Alkalinity (pH Level)	CPD	0-60%	13.2-13.25.	[175]
	CKD	0-60%	13.21-13.29.	[176]
	CKD	0-50%	12.1-12.6.	[177]
	Fly Ash	0-20%	12.7-12.8.	[178]
	GGBF slag	0-50%	11.57-12.24.	[171]
Electrical resistivity	Biochar	5%	increased by 1000%.	[174]
	Quartz	20%	increased by 3800%-7200%.	[179]
	limestone	20%		

¹ Performed according to DIN 1048

² Performed according to ASTM C1202

³ Performed according to NT Build 492

Although ACC technology positively enhances the durability of concrete and its porosity, making it less vulnerable to aggressive substances such as chlorides, another critical aspect

remains underexplored within this context is the chloride binding capacity of concrete subjected to ACC.

Generally, corrosion of steel in reinforced concrete, primarily caused by the infiltration of chloride ions, is a significant factor in the degradation of such structures, particularly in coastal settings or when exposed to deicing agents. As chloride ions enter the concrete, some remain free within the pore solutions and initiate corrosion of the steel when reaching a critical level. Conversely, the rest that are bound by cement hydration products can reduce the free chloride ion concentration and thus retarding corrosion risks [180,181]. It is widely known that chloride ions bound within the concrete are categorized into two types: physical binding, largely dependent on the specific surface area of C-S-H, and chemical binding, which involves reactions between chloride ions and components such as tricalcium aluminate (C_3A) or calcium monosulphoaluminate (Afm), resulting in the formation of Friedel's salt [180,182]. Yet, these bound chloride ions become unstable when the concrete undergoes weathering carbonation. [183] discovered a direct correlation between bound chloride ions and pH levels in carbonated pastes. Furthermore, [184] investigation on the chloride binding ability of pastes in natural carbonation conditions revealed that chemical binding ability was completely lost, while the physical one was only partially lost.

According to earlier findings presented in Table 6, concrete's resistance to chloride penetration is notably enhanced through ACC. These studies have extensively examined how the physical pore structure and chloride coefficient evolve during ACC. However, the effects of ACC on chloride binding capacities have received little attention. Unlike weathering carbonation, which affects reinforced concrete structures post-hydration, the impact of ACC on the microstructure and chemical composition of cement-based concrete typically occurs during the early hydration stages. Thus, more research is necessary to understand how ACC alters the chloride binding mechanisms. Additionally, chloride ions may be incorporated into concrete through the use of raw materials containing chloride, such as sea water or marine sand, during the mixing process, or from external sources during the operational life of the structures. Each method of chloride introduction has its unique binding dynamics in carbonated concrete, an area that remains inadequately explored and warrants further investigation.

While the influence of ACC on chloride binding mechanisms demands further investigation, another crucial property that remains largely unexplored is the fire resistance of ACC-treated concrete.

Concrete typically remains stable under inert conditions, and brief exposures to high temperatures minimally impact its initial performance [185]. Prolonged exposure, however, can drastically alter its chemical, physical, and mechanical characteristics. Such thermal stress often leads to cracking that degrades the material and compromises the structural serviceability [186,187]. Therefore, numerous studies have been undertaken to

examine how concrete behaves when subjected to elevated temperatures [188–191]. Despite this, the response of ACC-treated concrete to high temperatures remains unexplored and needs further research.

Effect of ACC on microstructural characteristics of cement-based concrete

The interaction between carbonation and hydration processes results in a distinct microstructural composition of cement-based concrete subjected to ACC compared to those undergoing conventional hydration. Analysis of the EDS spectrum, as illustrated in Figure 5(a) and (b), demonstrates an increase in calcium and carbon levels in ACC-treated samples, whereas silicon levels are reduced, suggesting the formation of CaCO_3 crystals during this process. SEM images, depicted in Figure 5(c1), (c2), and (c2), highlights that the primary constituents of ACC-treated samples are polycrystalline calcium carbonate (predominantly in the form of prismatic calcite) and partially decalcified C-S-H, forming a layered structure. The samples also showcase C-S-H and portlandite as core hydration products. Notably, the surfaces coated by CaCO_3 and decalcified C-S-H exhibit a compact structure to those coated by hydration products, with CaCO_3 serving to fill and refine the surface pores of the entire matrix [192,193]. Clearly, as depicted in Figure 5 (c3), the presence of dentate aragonite (a less stable variant of CaCO_3) increases with the increase of carbonation degree to a certain degree, a phenomenon observed on highly carbonated cement surfaces [194,195].

As illustrated in Figure 5(d1), (d2), and (d3), the primary hydration products in non-ACC-treated samples are C-S-H, portlandite, and ettringite. For ACC-treated samples, C-S-H presence in Figure 5(d2) indicates that cement hydration, even when partially carbonated, remains unaffected. Nevertheless, during ACC, formation is limited to calcite and aragonite crystals, and no C-S-H or portlandite is formed in Figure 5(c1) and (d3), showing how extensive carbonation can hinder further hydration and strength development.

In conclusion, CaCO_3 formation through ACC substantially enhances the densification and refinement of the pore structure in cement-based materials, as evidenced by improved microstructural characteristics.

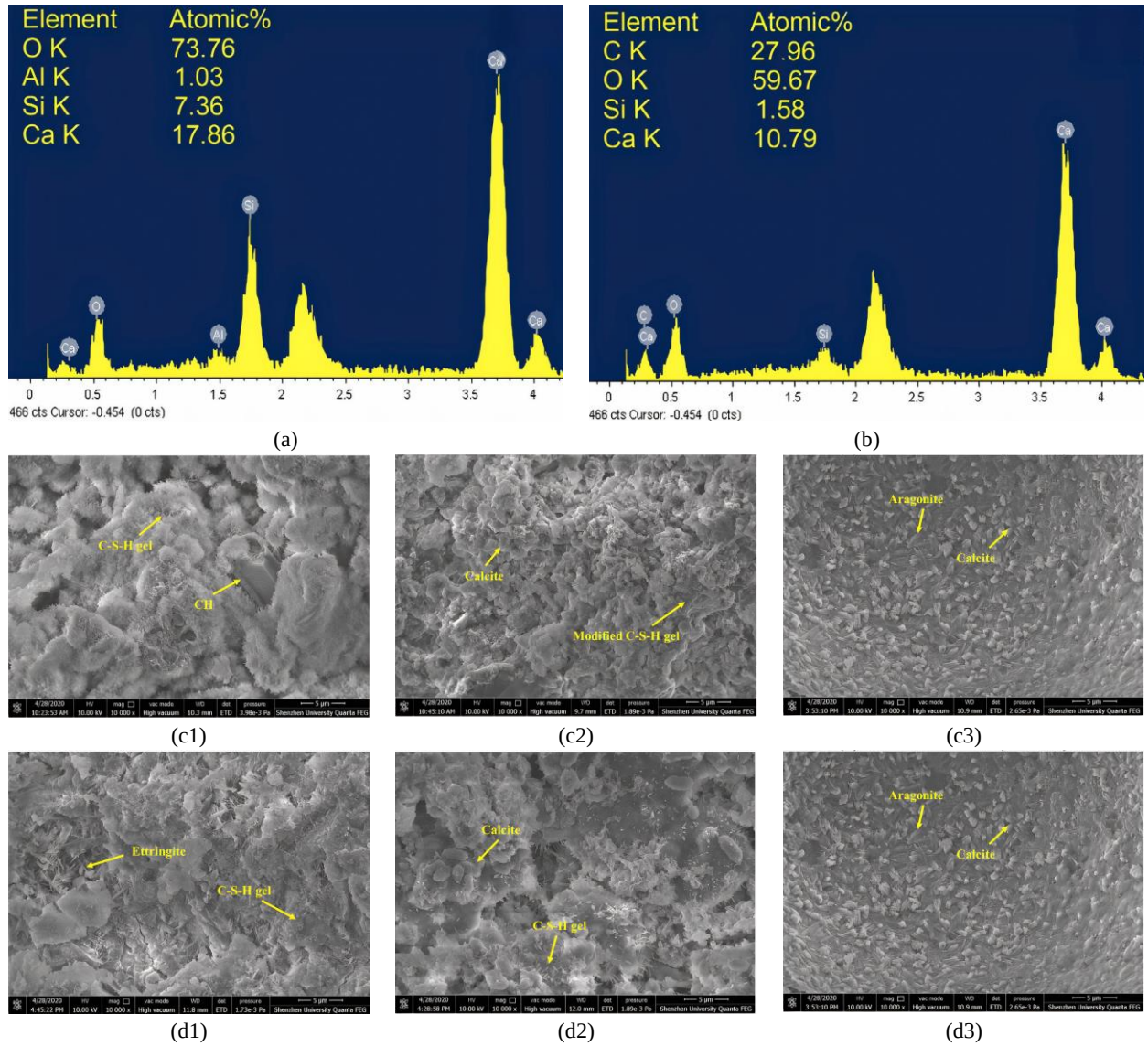


Figure 5: Microstructural characteristics of ACC-treated OPC-based concrete: (a) and (b) EDX spectra of ACC-treated and non-ACC-treated specimens; (c1), (c2), and (c3) SEM images of hydrated specimens underwent 0, 1, and 72 h ACC; (d1), (d2), and (d3) SEM images of 28 days hydrated specimens underwent 0, 1, and 72 h ACC (reused with permission from [137,196]).

2.2.4 Pre-carbonation method

Despite the advantages of ACC discussed previously, it has significant limitations. One major limitation is the slow diffusion of CO_2 into the concrete, as carbonates form and fill the pores of the concrete, limiting the efficacy of this approach [17]. Additionally, the need for early-stage concrete to be placed in a sealed chamber with high CO_2 concentrations confines the application of this method to precast concrete.

To address these challenges, a novel approach has been introduced by [18], which involves directly injecting CO_2 into fresh concrete mix as an admixture through a two-stage mixing process, pre-carbonation process [18]. Firstly, CO_2 is injected into a slurry of Ca-rich

cementitious material, followed by mixing with other components to produce concrete in the second stage (Figure 6).

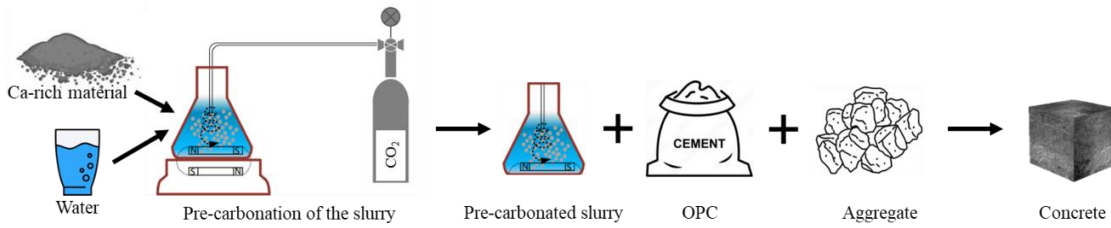


Figure 6: Pre-carbonation method (summarized from [18])

Slaked lime, used as Ca-rich materials, was mixed with water and at 1 and 3% (by weight of water). The slaked lime slurry was carbonated by injecting CO₂ into the slurry until full carbonation was achieved. Scanning electron microscopy (SEM) images shown in Figure 7 illustrate the precipitates from this carbonation process, revealing nano to sub-micro sized particles. Notably, particles from the 3% slurry (Figure 7(a)) are larger than those from the 1% slurry (Figure 7(b)), a difference attributed to the extended filtration time required for the 3% slurry, which allowed smaller particles more time to aggregate into larger formations.

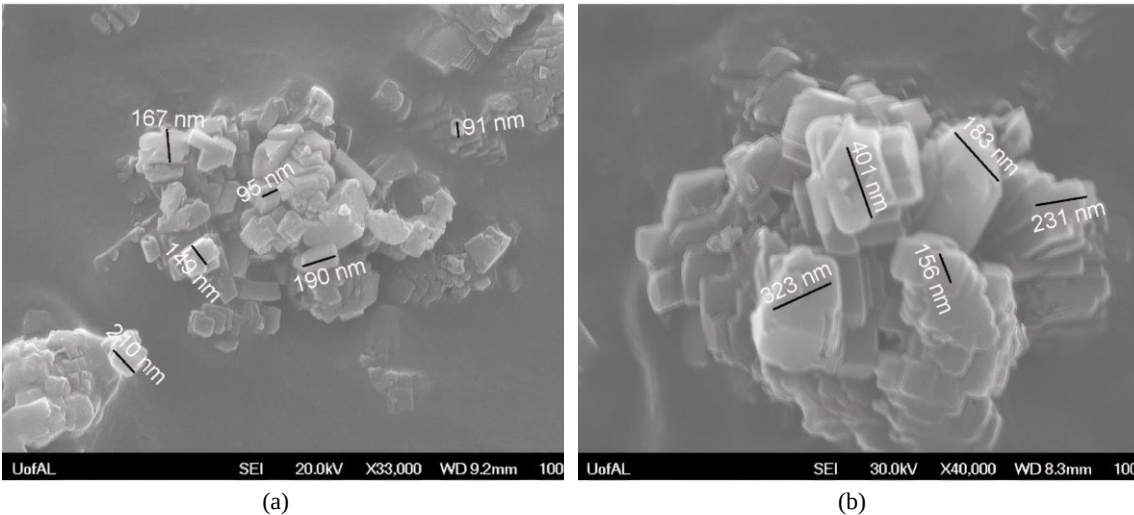


Figure 7: SEM image of precipitates form carbonated slaked lime slurries at: (a) 1%; (b) 3% (reused with permission from [18]).

The researchers found that the pre-carbonation approach significantly enhances the compressive strength of cement mortars compared to the control mix. The 28-days compressive strength of mix 1% and mix 3% exceeded that of the control by 16% and 13% respectively. This enhancement is supported by thermogravimetric analysis (TGA), which shows increased ettringite formation in all pre-carbonated samples as shown in Figure 8. Furthermore, TGA results reveal an increase in CH content in these samples. The weight losses from CH decomposition between 400 °C and 500 °C for the control, 1%, and 3% mixes reported as 3.59%, 3.63%, and 3.71%, respectively. The presence of more CH

suggests enhanced hydration of C3S in the pre-carbonated mixes indicating that the pre-carbonation method effectively promoted cement hydration. Additionally, the increased ettringite formation in the pre-carbonated mixes occupies more space compared to monosulfate, which helps maintain similar drying shrinkage levels as the control mix.

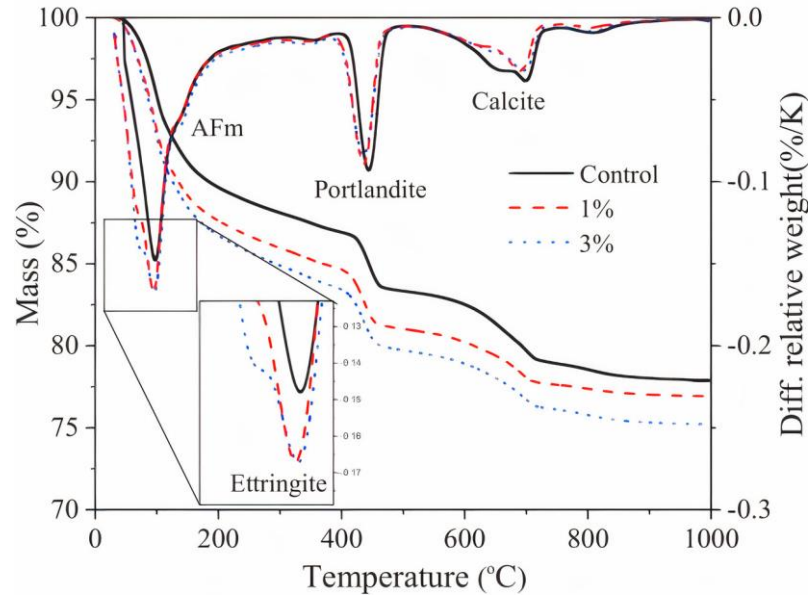


Figure 8: TGA results of 4-days OPC pastes incorporating pre-carbonated slaked lime slurry (reused with permission from [18]).

Despite these encouraging results, further research is necessary to understand the effect of this approach on the long-term properties of concrete, especially at higher replacement levels. Additionally, the lime manufacturing process, especially during calcination, is a major source of industrial CO_2 emissions. The carbonation of lime recaptures some CO_2 , but not enough to fully counterbalance the emissions generated during its production. Therefore, Ca-rich materials such as Class C fly ash and slag, can be a better sustainable alternative to replace lime in real-world applications of this carbonation technique. Moreover, the practicality of the carbonation process for in-situ applications is a significant obstacle.

2.2.5 Carbonation mixing

During the batching and mixing of concrete, CO_2 can be injected into concrete mixture, This leads to the formation of amorphous CaCO_3 , likely in a nanocrystalline form, which in turn speeds up cement reaction and boosts its compressive strength gain [197,198]. Although the quantity of CO_2 absorbed by the concrete is minimal, this technique could lower the amount of cement required, thereby reducing the carbon footprint associated with concrete production [197]. Another strategy involves capturing CO_2 in the residual slurry from concrete mixer and truck clean-up, and then using the carbonated slurry as an additive in new concrete production [199,200]. However, the capacity for CO_2 capture through this

method is limited and the availability of this material is only around 1 to 2.5% of the production of ready-mix concrete [201].

Figure 9 depicts a setup for used for mixing in the CO₂ [202]. It consists of a mixer and a cover. CO₂ is injected continuously at a rate of 5 L/min into the bowl through the seal cover's inlet and released through the outlet. To prevent emissions, supersaturated sodium hydroxide solution is utilized for the capture of residual CO₂. Two mixing methods are used: carbonation mixing (240 s) and hybrid mixing. The hybrid method involved two steps: CO₂ mixing (240 s) and subsequent air mixing (120 s) without the cover. Carbonation using either of the two methods is followed by only carbonation curing for 60 min.

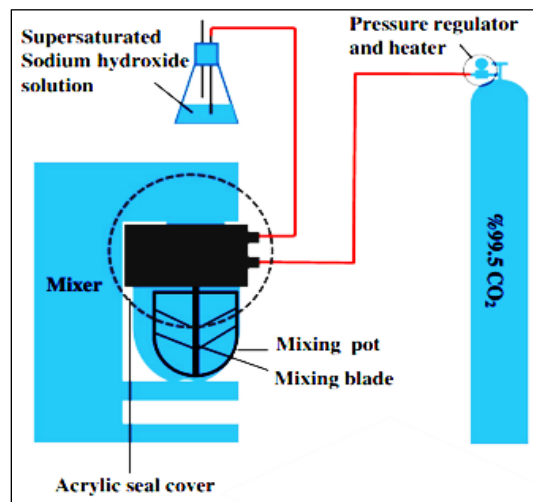


Figure 9: CO₂ mixing setup (reused with permission from [202]).

As shown in Figure 10, the researchers [202] reported that the carbonation mixing (CM) results in a reduction in cement paste strength. The observed phenomenon can be attributed to the prompt development of carbonation products on the surface of cement particles during the initial hydration stages, which could potentially impact the inter-particle cohesion Figure 11(a). The strength is enhanced by hybrid mixing (HM) due to the disruption of the carbonate encasing through secondary mixing Figure 11(b). However, strength remains significantly lower than with normal air mixing followed by carbonation curing (NC) Figure 10. The implementation of this technique will facilitate the ability of ready-mix concrete to apprehend and retain carbon dioxide, resulting in a noteworthy augmentation of the carbon sequestration potential of concrete products. Nevertheless, additional investigation is required regarding the decrease in strength that is linked to it.

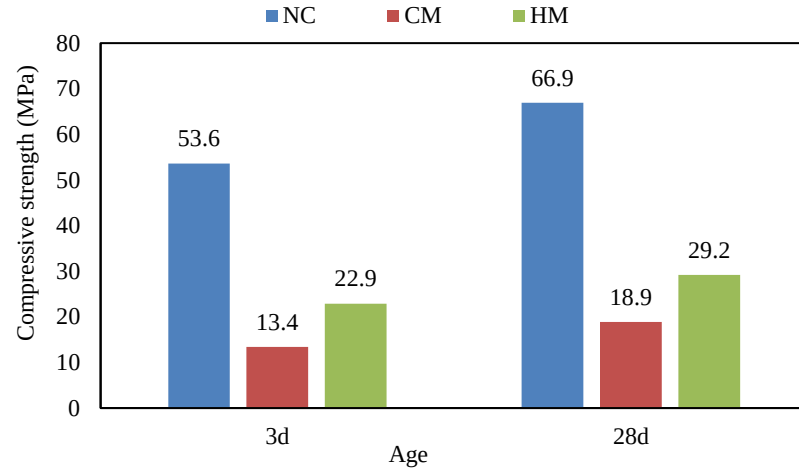


Figure 10: Compressive strength for carbonation mixing, hybrid mixing and normal air mixing with carbonation curing [202].

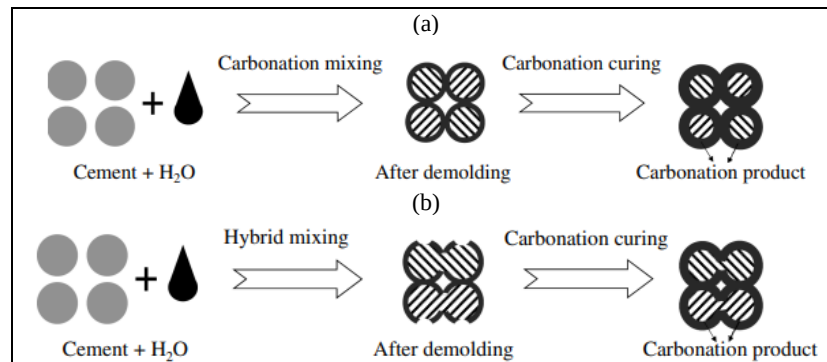


Figure 11: Schematic of microstructure development: (a) carbonation mixing, (b) hybrid mixing (reused with permission [202])

In [203,204], a groundbreaking retrofit solution designed for CO₂ sequestration in ready-mix concrete production, ensuring compatibility with established manufacturing protocols. During the batching phase, CO₂ was injected, with a metering system delivering a regulated amount of pressurized liquid CO₂ to a discharge outlet. The addition of CO₂ was similar to the addition of an admixture, meaning no impact on batching or loading cycle time. Once CO₂ came into contact with concrete, changed the state into a solid state, similar to dry ice or snow, due to a significant drop in pressure. Direct injection into the central mixer before truck loading ensured rapid reaction of CO₂ with calcium ions from the hydrating cement, forming CaCO₃. Solid CO₂ was sequestered within the concrete matrix without leaving any gaseous residues. It was found that while removing 7% of the binder from concrete reduces its strength, adding CO₂ can effectively restore its compressive strength (Figure 12) [205].

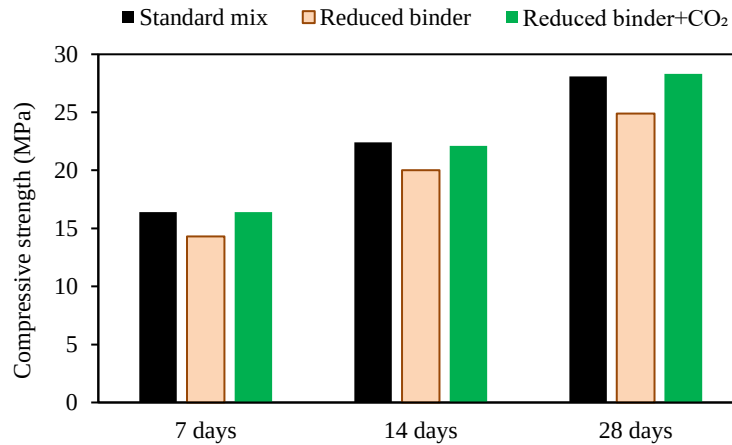


Figure 12: Effect of injecting CO₂ into concrete mix produced with 7% binder reduction [41]

Later, this technology was commercialized as CarbonCure [206], marking a significant advancement in the concrete industry's efforts towards sustainability. CarbonCure's goal now is to achieve a reduction of 550 million tons of CO₂ emissions annually by 2030. Already, numerous CarbonCure-ready mix concrete manufacturers in Canada are making substantial contributions towards this target, producing 25 million m³ of concrete and utilizing 8.5 million tons of CO₂, thereby preventing 354 million tons of CO₂ emissions [207]. Additionally, a life cycle assessment (LCA) of CarbonCure technology has shown it to reduce net CO₂ emissions by approximately 18 kg/m³ of concrete, highlighting the substantial environmental benefits of this pioneering approach [205].

Despite the promising results of CarbonCure technology, it still remains in the early stages of investigation. Therefore, establishing standards and protocols for the effective use of this technology would greatly benefit ongoing research and practical implementations. In addition, further investigation is crucial to comprehensively understand the long-term impacts of this technology on the durability of concrete. Furthermore, while this technology is currently tailored primarily for the ready-mix concrete sector, there is a clear need to refine the process to make it more user-friendly for in-situ applications.

2.3 Ecological footprint of accelerated CO₂ sequestration

The successful implementation of mineral carbon dioxide sequestration necessitates the careful evaluation of the environmental ramifications associated with the accelerated carbonation process, which typically entails substantial energy expenditure. Conducting a life cycle assessment (LCA) is a feasible approach to evaluate this impact, as reported by several researchers [208–211]. The ecological impact of producing carbonated materials is often assessed using the Global Warming Potential (GWP). The GWP quantifies and compares the climate impact of various substances by measuring the heat trapped by a

greenhouse gas over a specific period (typically 100 years), relative to CO₂. For a material, GWP is expressed as kilograms of CO₂ equivalent per kilogram of the material (kg CO₂ eq./kg), indicating the emissions of CO₂ or its equivalents that would have the same global warming impact as one kilogram of the material assessed over a standard time frame [212,213].

Figure 13 presents the GWP data pertaining to the manufacturing of diverse construction materials, excluding accelerated carbonation process. The data indicates that the manufacture of one kilogram of cement typically results in the emission of 0.95 kilograms of carbon dioxide [209,214–216], which is notably greater than the GWP figures for other materials [217,218].

While using slag and fly ash as substitutes for OPC is observed as environmentally beneficial, GWP information, after carbonation is carried out, for these alternatives remains limited.

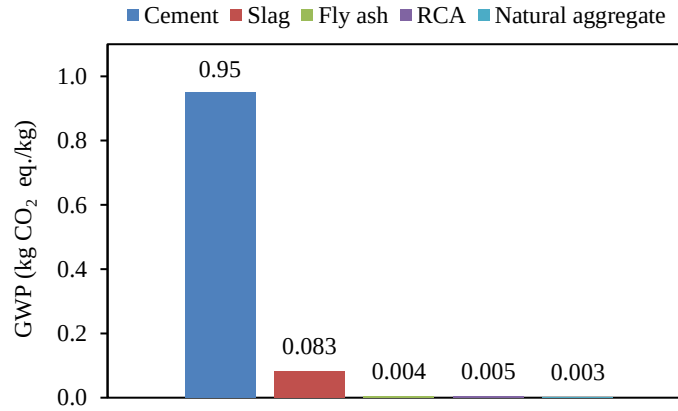


Figure 13: Comparative GWP values of various construction materials [209,214–218].

The production of carbonated materials is associated with a significant rise in CO₂ emissions, primarily due to the high electricity consumption involved in the accelerated carbonation process. The consumption of electricity is significantly correlated with carbonation parameters, including but not limited to duration, pressure, temperature, and infrastructure. Unfortunately, currently there is an absence of detailed research and dependable empirical evidence concerning the GWP of the industrial waste materials that have undergone accelerated carbonation process. Therefore, the ensuing discourse, as depicted in Figure 14, centers primarily on GWP assessment typically for slag and fly ash in the context of carbonation process.

According to Baciocchi et al. [219], the electricity required for the carbonation of 1 ton of CO₂ via aqueous carbonation of slag amounts to 493 kWh. According to the studies reported by several researchers [220–223], the mean GWP of electricity (GWP-E) is 0.5 kg CO₂ eq./kWh. Consequently, the GWP of carbonated slag has been calculated to be

0.101 kg CO₂ eq./kg. The calculated CO₂ uptake capacity was determined to be 0.403 kg CO₂ /kg. Because the calculated GWP-E value is lower than that of the uptake, therefore, the net GWP for slag is negative.

Based on prior computations, studies [224,225] have shown that the electrical energy usage required for carbonating 1 ton of CO₂ via aqueous carbonation of slag and fly ash were 447 kWh and 293 kWh, respectively. Correspondingly, the GWP value of treated slag and fly ash are 0.045 kg CO₂ eq./kg and 0.004 kg CO₂ eq./kg, respectively. The computed CO₂ uptake capacities were 0.2 kg CO₂ /kg for slag and 0.027 kg CO₂ /kg for fly ash. Therefore, the net GWP for slag and fly ash are negative.

The GWP of gas-solid carbonation process applied to slag was approximated to be 0.21 kg CO₂ eq./kg (Figure 14 (b) [95]). The measured CO₂ absorption capacity was approximately 0.089 kg CO₂ /kg slag. As a result, the net GWP was found to be positive (CO₂ capture is lower than CO₂ emissions).

It is noteworthy that the GWP values specified above are predominantly derived from laboratory experiments due to the scarcity of information obtained from industrial-scale carbonation system. Furthermore, the assessment of the GWP may be incomplete as it fails to sufficiently account for the energy usage associated with materials transportation. The estimated values of GWP may potentially exhibit an underestimation of the factual greenhouse gas emissions in real-world scenarios.

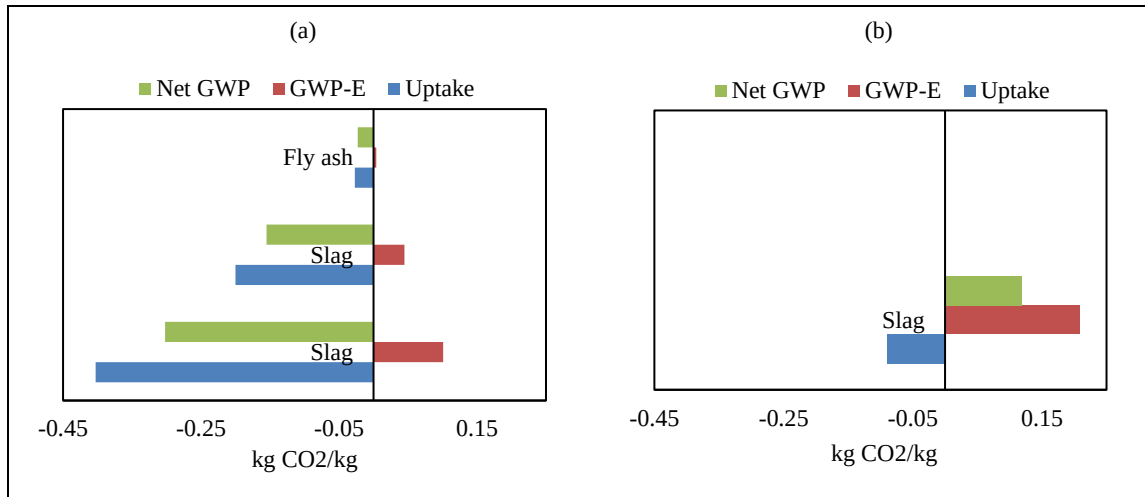


Figure 14: The net GWP of carbonating: (a) slag and fly ash through aqueous carbonation [219–225], (b) slag through gas-solid carbonation [95]

2.4 industrial outlook, challenges and prospects

In the past twenty years, several companies have dedicated resources to creating patented solutions aimed to lower carbon emissions from cement/concrete production, while advancing the production of building materials through different carbon capture and utilization (CCU) methods. Besides CarbonCure discussed as carbonation mixing

technology in 2.2.5, this section discusses these commercialized CCU technologies. Additionally, Table 7 provides a summary of the benefits and drawbacks of these technologies, along with the research gaps that need to be addressed.

2.4.1 Solidia

Established in 2008 in Piscataway, New Jersey, USA, Solidia Technologies specializes in eco-friendly solutions by utilizing CO₂ to create sustainable building and construction materials [226–229]. The company offers two main products, Solidia Cement and Solidia Concrete, both of which generate significantly lower CO₂ emissions compared to traditional building materials. Solidia Cement, an alternative to hydraulic cement, is predominantly made from wollastonite and rankinite rather than the traditional alite and belite found in OPC [230]. This reduction in lime content per unit of cement means less limestone needs to be calcined, thus cutting emissions by about 30% [231].

Solidia Concrete is distinguished by a unique patented curing process that utilizes a mixture of fine and coarse aggregates, CO₂, and Solidia Cement. Unlike OPC that reacts with water, Solidia Cement reacts exclusively with CO₂ during the curing phase to form a stable form of calcium carbonate. This carbonation process produces calcite polymorphs of CaCO₃ and silica gel, which are essential for the concrete's strength development [230]. Solidia Concrete offers the advantage of curing in just 24 hours, which translates into cost savings, reduced time, and less required storage space for cement and concrete manufacturers [232].

2.4.2 Carbstone

In the 1990s, slag was abandoned after being extracted from ore. Today, it is transformed into bricks through a process of carbonation. Steel slag, rich in magnesium and calcium silicates, is converted into products when combined with captured CO₂ [233]. Annually, the steel industry in Belgium produces about 500,000 tons of steel slag [234]. In 2004, the recycling company Orbix found that a mixture of CO₂ and water could solidify the steel slag particles [235].

The carbonation process includes three phases: initial handling of slag; shaping building blocks using a hydraulic press at pressures ranging from 7 to 60 MPa to achieve the desired porosity; and the absorption of CO₂ into the slag under elevated temperatures and pressures [235,236]. During this chemical reaction, CO₂ reacts with calcium silicate to produce calcium carbonate, serving as a sustainable replacement for cement in building blocks and effectively trapping CO₂ for extended periods [237]. Introduced in 2006 as Carbstone, this building material offers a more environmentally friendly alternative to traditional concrete blocks, with a reduced carbon footprint of 0.2 CO₂ kg/kg slag compared to standard concrete blocks [238].

2.4.3 Calera

Calera, a company based in Los Gatos, has developed an innovative carbon capture technology that transforms mineral carbonate into a stable carbonate slurry, suitable for producing cement and other building materials [239,240]. In this process, flue gas interacts with alkaline Ca/Mg-rich solutions to produce thermodynamically stable calcium carbonates. These carbonates are then utilized as SCM. This method not only reduces carbon emissions relative to conventional cement production but also creates economic benefits [41].

2.4.4 Carbon8

In 2006, Carbon8 Systems Ltd. was established by the University of Greenwich (UK) to bring Accelerated Carbonation Technology (ACT) to the market [241,242]. This technology creates synthetic limestone by reacting CO₂ with varied industrial by-products (such as cement kiln dust and steel slags). Initially, alkaline solid waste is mixed with CO₂ and water. Then, during the mixing stage, binders and fillers are incorporated with the carbonated waste. The mixture is then fed into a pelletizer where CO₂ is introduced to accelerate the hydration process, resulting in the formation of rounded aggregates [243]. By 2012, ACT had been commercialized as Carbon8 Aggregates through an investment from Grundon Waste Management, with an annual production exceeding 150,000 tons. This process has the capacity to sequester more than 1 million tons of CO₂ annually using just 20% of the waste available in Europe [244]. Additionally, each ton of Carbon8 aggregate produced conserves 1.3 tons of natural aggregate and diverts 0.45 tons of waste from landfill sites [245,246].

Table 7: Benefits/drawbacks and research gaps of various CCU technologies (summarized from [230–232,235,236,238–240,243,247])

Technology	Final product	Benefits	Drawbacks	Research gap
CarbonCure	Concrete	Reduces 7% of the binder for same conventional concrete's strength.	- Only 10% CO₂ uptake (by cement weight) can be achieved. - Only applicable for precast concrete.	Effects on long-term concrete properties.
Solidia	Cement/concrete	- Captures 0.3 CO₂ kg/kg of cement in concrete. - Reduces water consumption. - Achieves 28 d strength in 24 hr.	Only applicable for precast concrete.	Comprehensive microstructure, mechanical, durability, LCA and scalability of technology.
Carbstone	Blocks	- Steel slag is recyclable. - Sequesters 0.2 CO₂ kg/kg of slag. - More CO₂ is sequestered than released.	Higher energy consumption which leads to CO₂ emissions.	- adaptation to various industrial waste streams. - LCA.

Calera	SCM	• Reduces OPC content in concrete. • Comparable strength development at low replacement levels.	• Higher energy consumption which leads to CO₂ emissions.	• Effects on microstructure, mechanical and durability properties of concrete. • LCA.
Carbon8	aggregate	• Transforms industrial wastes into usable materials. • Preserves natural aggregate resources.	• depends on consistent waste quality and supply.	• Effects on mechanical and durability properties of concrete. • LCA.

CHAPTER 3

DEVELOPMENT OF OPC PASTES INCORPORATING CARBONATED CALCIUM-RICH MATERIALS

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3.1 Abstract

Accelerated carbonation treatment is gaining significant attention as a viable means to reduce the carbon footprint in the concrete industry. However, the highly concentrated CO₂ environment required to carbonate concrete limits its application to pre-cast concrete. Recent studies attempted to address this limitation by incorporating CO₂ directly into the fresh concrete mixtures but their approaches remain challenging for on-site concrete mixing. To overcome this, the current study proposes introducing mineral carbonation into fresh concrete mixtures in three steps. Selecting hydrated lime and ordinary Portland cement (OPC) as calcium-rich materials (CRMs), this study proposes injecting CO₂ into slurries of these CRMs, powdering the carbonation products, and then incorporating them into concrete mixtures. Calcite was found as the dominant component in the carbonated CRMs, as evidenced by distinct calcite peaks observed in both thermogravimetric analysis (TGA) profiles, X-ray Diffraction (XRD) spectra, and morphology changes noted in scanning electron micrographs. The incorporation of carbonated CRMs in paste mixtures at OPC replacement levels of up to 15% sufficiently preserved the hydration products, as revealed by TGA and XRD. This resulted in enhanced early-age strength and sustaining above 95% of the compressive strength after 28 days, suggesting that the proposed carbonation method offers a path to sustainability in concrete production without significantly compromising mechanical performance.

Keywords: carbon dioxide, mineral carbonation, calcite, cement, hydration product.

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3.2 Introduction

The cement and concrete industries have been identified as significant contributors to global carbon dioxide (CO₂) emissions, accounting for approximately 8.0% of the world's total emissions [248,249]. The cement and concrete products market, valued at \$370.1 billion in 2019, is anticipated to expand rapidly, with projections suggesting a compound annual growth rate of 12% from 2021 and a global cement production of 5 billion tons by 2030 [248]. This growth is likely to result in cement and concrete production representing over 10.0% of global CO₂ emissions between 2030 and 2050 [248].

While manufacturing ordinary Portland cement clinker releases CO₂, the hydrated clinker in hardened cement gradually reabsorbs CO₂ into concrete products through a slow natural chemical process. However, this reabsorption is inefficient, potentially taking centuries to neutralize most of the CO₂ released during clinker manufacture [14,15]. Consequently, there is growing concern to balance the high global demand for cement-based materials with the need to decrease greenhouse gas emissions. Implementing effective strategies is thus vital for the concrete industry's sustainable evolution [248,249]. One strategy involves substituting cement clinker with supplementary cementitious materials [250–252], as clinker production is responsible for about 85% of concrete's total CO₂ emissions [253]. However, this substitution strategy alone falls short of achieving global CO₂ reduction goals.

To further mitigate CO₂ emissions, technologies like carbon capture, utilization, and storage (CCUS) are being considered. CCUS involves capturing CO₂ from stationary sources and then utilizing or storing it in various forms [254]. Accelerated carbonation curing (ACC) is a CCUS technique that involves exposing early-age concrete to high concentrations of CO₂, allowing the gas to react with the fresh concrete and transform it into solid calcium carbonates (CaCO₃). This not only enhances the concrete's strength [154,159,162–168] but also reduces water penetrability [167] as well as chloride permeability [167,173].

Another CCUS technique is mineral carbonation, which involves storing carbon dioxide in a stable carbonate form, such as CaCO₃ and MgCO₃. This is achieved by accelerating the natural process of carbonating Ca- and Mg-rich materials, resulting in the permanent storage of CO₂. Mineral carbonation has been conducted using alkaline solid waste materials – such as steel slag [53], recycled concrete aggregates [59–61,73–75], and fly ash [56–58,71,72] – instead of natural resources to save nature and reduce energy consumption in the grinding and extraction of naturally occurring carbonatable minerals. These materials are favored due to their high reaction rates, low energy requirements, and efficient carbon

conversion compared to natural minerals [48,49]. Despite its potential, using these carbonated materials as a substitute for Portland cement in concrete has been explored in only a few studies.

A study attempted to incorporate mineral carbonation into cement mortar by pre-carbonating lime as a calcium-rich material (CRM) before mixing the wet carbonate with other ingredients [18]. The findings revealed that using 1% carbonated lime in the mortar improved its compressive and flexural strength by 16% and 5%, respectively, and slightly reduced 28-day drying shrinkage. Despite these encouraging results, further research is necessary to generate additional data, including the effects of higher replacement levels of carbonated lime on the characteristics of cementitious composites and the environmental advantage of its use as cement replacement. Additionally, the practicality of the carbonation and utility process proposed by the referenced study for in-situ applications is a significant obstacle, necessitating modifications.

Similarly, previous studies have introduced CO₂ during the concrete mixing stage, a method known as carbonation mixing [197,198,202]. In this method, gaseous CO₂ is continuously injected at a constant rate into the mixer. However, this method significantly reduced the strength of cement paste by 56% [202]. In contrast, a different approach utilized liquid CO₂ instead of gaseous CO₂ [203,204], which has been commercialized as CarbonCure, a retrofit technology for ready-mix concrete plants. In this carbonation mixing alternative, liquid CO₂ is accurately metered and injected during the batching phase of ready-mix concrete. Upon contact with the hydrating cement, CO₂ changed the state into a solid state, similar to dry ice or snow, due to a significant drop in pressure. CO₂ rapidly reacts with calcium ions to form CaCO₃, which is then permanently embedded within the concrete matrix. It was found that while removing 7% of the binder from concrete reduces its strength, adding CO₂ can effectively restore its compressive strength [205]. However, despite the carbonation mixing benefits, adapting it for on-site concrete mixing is difficult due to equipment challenges and the need to transport and store liquid CO₂ on-site. Therefore, there is a need for a modified approach to work around these challenges.

To address these identified research needs, the present study proposes preparing dry powders of carbonated CRMs, which can then be bagged, stored, and distributed for use in construction applications in a similar way to other construction materials. Specifically, this study considered the use of dry carbonated forms of two CRMs, introducing mineral carbonation into the fresh OPC-based mixtures in three stages. The first stage involves the introduction of CO₂ into a slurry of a CRM, followed by filtration, drying, and declumping of the carbonated slurry. The final stage involves the incorporation of the powdered product into concrete. This protocol is particularly suited for in-situ applications as carbonation occurs ex-situ, allowing the powdered product to be stored and used as a cement substitute.

Aiming to explore this proposed carbonation mixing protocol, this paper reports a study conducted on the production and characterization of two different carbonated CRMs. The study examines the effects of incorporating carbonated CRMs at various levels on the hydration kinetics, compressive strength, and microstructure of cement paste.

3.3 Experimental Program

3.3.1 Materials

Two primary materials were considered in this study as calcium-rich materials (CRMs): industrial hydrated lime and ordinary Portland cement (OPC), complying with ASTM C 150. Industrial CO₂ gas with 99.9% purity was used for carrying out the carbonation process. Limestone powder (LSP) was used as a natural calcium carbonate (CaCO₃) benchmark to study the effects of incorporating the chemically precipitated CaCO₃ resulting from the carbonation of CRMs. As obtained by X-ray fluorescence (XRF) analysis, the chemical compositions of primary study materials are presented in Table 8.

Table 8: Chemical composition (m/m %) of lime, OPC, and LSP.

Oxide	Lime	OPC	LSP
CaO	68.01	64.64	32.66
SiO ₂	0.29	17.30	7.27
Al ₂ O ₃	0.64	4.00	1.77
Fe ₂ O ₃	0.13	3.19	1.21
SO ₃	0.09	1.65	0.15
K ₂ O	0.07	1.04	0.82
P ₂ O ₅	0.55	0.67	0.42
TiO ₂	0.02	0.24	0.16

3.3.2 Production of carbonated CRMs

Six different slurries were produced by mixing samples of each CRM with 500 ml deionized water in a 1-liter flask at varying concentrations (5%, 10%, and 15% by weight of water). Each CRM slurry was left to hydrate for 1 hour after which the hydrated slurry was carbonated by directly injecting CO₂ gas through the slurry at a flow rate of 6 L/min. A magnetic stirrer was employed to agitate the slurry during carbonation. To ensure a high degree of carbonation at a minimum duration, the pH level of the carbonating slurry was continually monitored at 5-minute intervals. The carbonation process was terminated when the pH attained a steady state.

During this process, CO₂ initially combines with water to create carbonic acid (H₂CO₃). Subsequently, carbonic acid dissociates into positively charged hydrogen ions (H⁺) and negatively charged carbonate ions (CO₃²⁻). These ions react with calcium ions (Ca²⁺)

released from CRM in solution [41,62–64]. As a result, CaCO_3 precipitates as nano to sub-micrometer particles.

The carbonated CRM was recovered from the slurry via vacuum filtration (using a Grade 3 filter paper with 5 μm particle retention) and dried in an oven at 40°C until a stable weight was attained. Finally, the resulting carbonate was pulverized using a low-power blender to disperse the clumped dry mass into a powdery material. The whole process of preparing a powdery carbonated CRM is depicted in Figure 15.

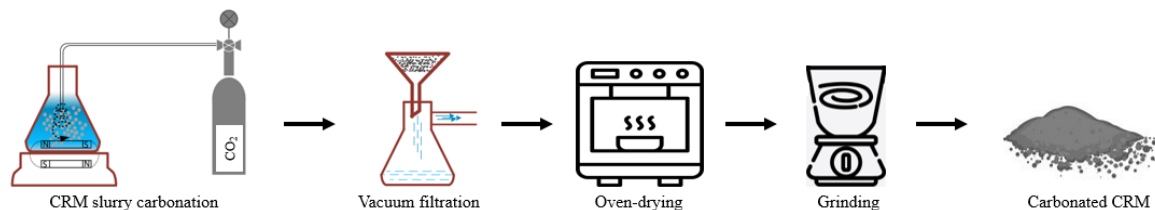


Figure 15: Production of carbonated CRMs.

3.3.3 Material characterization

The particle size distribution (PSD) of the raw and pulverized carbonated CRMs was obtained using an Anton Paar particle size analyzer in a wet medium of ethanol. Water was intentionally avoided as a dispersant in order to prevent any potential chemical reactions with the CRMs that could corrupt the PSD results. The specific gravities of raw and carbonated CRMs were determined using a 50-ml pycnometer filled with ethanol. Thermogravimetric analysis (TGA) of fresh and carbonated CRMs was carried out by heating materials from 30 °C to 1000 °C at 10 °C per min using Perkin Elmer STA 6000 to quantify their CaCO_3 contents. X-ray diffraction (XRD) and scanning electron microscopy (SEM) were employed to examine the materials' mineralogy and surface morphology, respectively. The XRD analysis was carried out using a Rigaku Ultima IV diffractometer, while SEM analysis was performed on JEOL 5800LV- Tungsten microscope. For these analyses, a total of 10 g of fresh/carbonated CRMs passing ASTM sieve #200 was used. In addition, the benchmark LSP was analyzed along with the carbonated CRMs.

3.3.4 CO_2 uptake capacity and carbonation efficiency

The carbonation of cement products serves two primary objectives in the concrete industry: reduction of carbon footprint and enhancement of the construction materials' performance. These objectives are closely linked to essential factors: the capacities of carbonatable materials to absorb CO_2 and carbonation efficiency (or degree of carbonation) [93,94,98,99].

In this study, the theoretical maximum CO₂ uptake (CO_{2-th}) was calculated using Steinour's formula presented in Eq. (30), based on the chemical composition of CRMs in terms of weight percentage, as reported in previous studies [255–258]. The actual CO₂ uptake (CO_{2-actual}) was determined using TGA.

$$\text{CO}_{2\text{-th}} = 0.785 (\text{CaO} - 0.56 \text{CaCO}_3 - 0.7 \text{SO}_3) + 1.091 \text{MgO} + 0.71 \text{Na}_2\text{O} + 0.468 \text{K}_2\text{O} \quad (30)$$

Eq. (31) was used to calculate the carbonation efficiency (η), representing the amount of CO₂ successfully sequestered by a material compared to its theoretical sequestration capacity.

$$\eta (\%) = \frac{\text{CO}_{2\text{-actual}}}{\text{CO}_{2\text{-th}}} * 100 \quad (31)$$

3.3.5 Preparation and characterization of OPC-substituted pastes

Calorimetry

An isothermal calorimetry test was conducted to examine the effect of replacing OPC with carbonated OPC (CC), carbonated lime (CL), and LSP at replacement levels of 15%, 30%, and 45% on the hydration process of cement paste. A water-to-cement ratio (w/c) of 0.5 was maintained for all paste mixtures. To eliminate the influence of temperature fluctuations on hydration, all specimens were prepared at the same initial mixing temperature of about 23 °C. The control cement paste specimen was made by mixing 50 g of cement with 25 g of deionized water for 2 minutes before placing in the calorimeter for 72 hours.

Preparation of paste samples

Cubical paste specimens, measuring 25 × 25 × 25 mm, were produced using paste mixtures incorporating CC, CL, and LSP, each at 15%, 30%, and 45% replacement levels and w/c of 0.5. After 24 hours, the specimens were demolded and cured under a damp burlap at 23°C.

Microstructure analysis

After 28 days of curing, the paste samples were crushed, and the hydration was stopped using isopropanol alcohol (IPA) as described elsewhere [259]. Following hydration stoppage, the paste samples were powdered and then examined by TGA and XRD. Another set of paste samples was sliced, and the hydration was stopped by immersing the slices in IPA for 5 days. During the first 24 hours, IPA was replaced every 8 hours and then every 24 hours. The sliced paste samples were examined by SEM.

Compressive strength

The compressive strength of paste specimens was evaluated at 3, 14, and 28 days. The compressive strength at each testing age was determined as the mean value of five identical replicate specimens.

3.4 Results and Discussions

3.4.1 Carbonation behavior of CRMs

Figure 16 illustrates the pH evolution of CRM slurries during the carbonation process. Each CRM slurry initially exhibits high alkalinity; however, the carbonic acid formed by injected CO_2 reacts with $\text{Ca}(\text{OH})_2$ in the slurry, forming CaCO_3 and consequently reduces the slurry's alkalinity. The pH eventually stabilizes, indicating carbonation completion. Generally, the time required for pH to stabilize increased with CRM concentrations in the slurry due to the greater quantity of $\text{Ca}(\text{OH})_2$ available for reaction at the same CO_2 flow rate. Furthermore, it was observed that the pH of OPC slurry stabilized more rapidly than that of lime at all concentrations. This accelerated stabilization in OPC slurries can be attributed to the exothermic heat released during cement hydration, which enhances the carbonation reaction rate [55,57]. Additionally, the amount of portlandite available in 1-hr hydrated OPC is lower compared to hydrated lime.

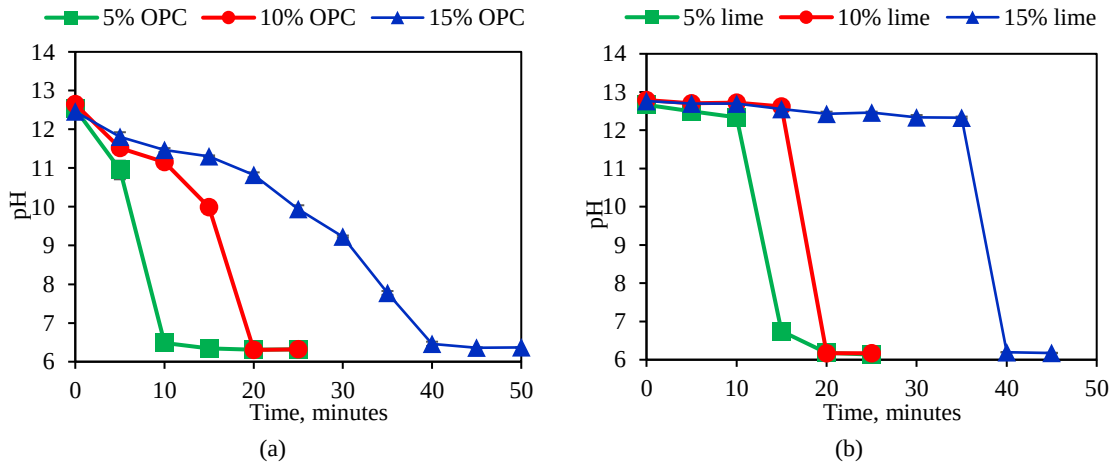


Figure 16: pH evolution during carbonation of (a) OPC and (b) lime slurries.

3.4.2 Characteristics of carbonated CRMs

Physical characteristics

Figure 18 and Figure 19a show the PSD and mean particle size of the fresh and carbonated CRMs, respectively. The data clearly indicates that carbonated CRMs exhibit a finer PSD when compared to their fresh counterparts, resulting in the mean size reduction of 29.4 and 14% in carbonated OPC and lime, respectively. This observation can be primarily ascribed

to the grinding process employed to declump the carbonated materials. Although its primary purpose was to break the dried material clumps of the carbonated CRMs into powder, the grinding process is expected to reduce the materials' particle sizes. The carbonated CRM particles are composed of loose outer and inner CaCO_3 particles that easily break down into finer particles under the mechanical energy imparted by the blender, as depicted in Figure 17. Consequently, the increased surface area of the finer carbonated CRMs enhances the reactivity of the materials.

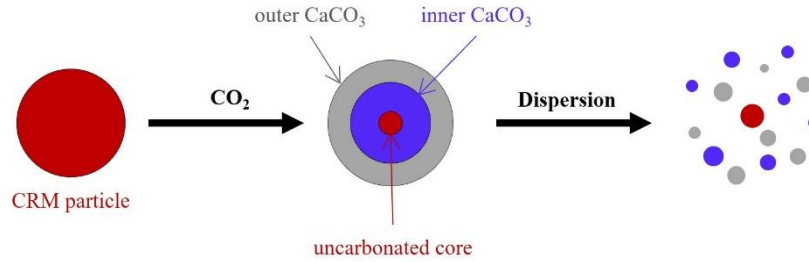


Figure 17: Morphology transformation mechanism of CRM particle.

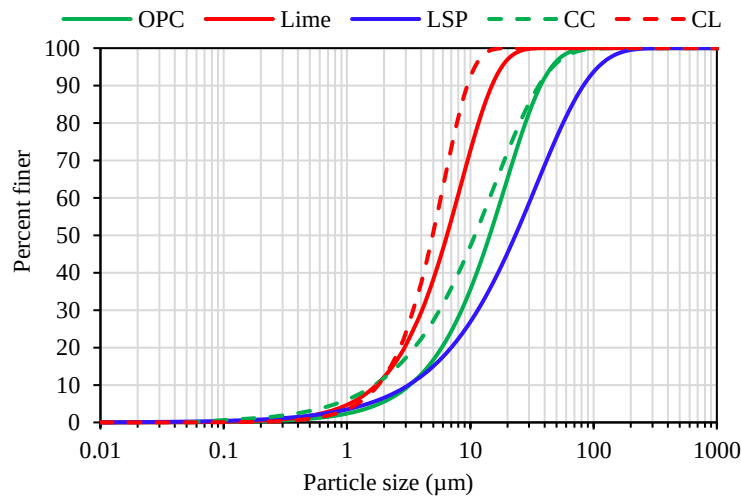


Figure 18: Particle size distribution of the raw and carbonated CRMs.

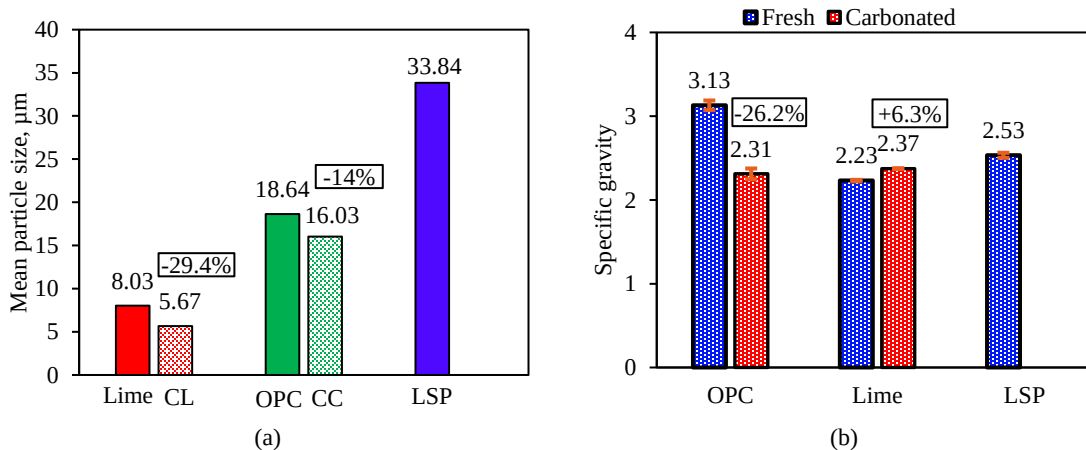


Figure 19: Effect of carbonation on (a) mean particle sizes and (b) specific gravity of CRMs.

Figure 19b shows the specific gravity (SG) of raw and carbonated CRMs. The data distinctly shows that CC exhibited a reduction in SG by 26.2% relative to its uncarbonated counterpart. This decrease is attributed to the formation of CaCO_3 , which has a lower density than raw OPC minerals [260], effectively reducing the overall SG. On the other hand, the SG of CL increased by 6.3% compared to raw calcium hydroxide. This increase is attributed to the higher density of CaCO_3 formed during carbonation [260,261], which elevates the overall SG.

Mineralogical characteristics

Figure 20 presents the DTG profiles of the fresh and carbonated CRMs, describing the transformations accompanying the thermal decomposition of carbonated lime and OPC phases. The results reveal a pronounced DTG peak in the temperature range of 400°C to 500 °C on the hydrated lime curve, signifying the thermal decomposition of portlandite. The mineral carbonate benchmark material LSP is principally composed of calcite, the most thermally stable CaCO_3 polymorph [262,263]. The LSP decomposition profile exhibits its characteristic high thermal stability indicated by higher decomposition temperatures, between 700 °C and 800 °C.

Considering the DTG profiles of CL, results indicate that the slurry solid content, resulting in different carbonation speeds (Figure 16), has minor effects on the thermal stability (roughly indicating degrees of crystallinity) of the precipitated CaCO_3 polymorphs. Further, comparing the DTG profiles of the CL with that of LSP, it can be noted that the CaCO_3 precipitated in CL exhibits thermal stabilities close to the calcite in LSP, albeit with a slight backward shift in peak temperature. It may then be inferred that calcite is the dominant CaCO_3 polymorph precipitated in CL.

In contrast, CC exhibits a less intense peak in the same temperature range, implying a lower CaCO_3 content. This observation is consistent with the expected lower free Ca^{2+} concentration in 1-hr hydrated OPC, as free Ca^{2+} from lime and C-S-H are the primary reacting species in the carbonation reaction leading to CaCO_3 formation. Further, the closeness of the CC's decomposition profile to LSP's also suggests calcite as the dominant carbonation product in CC.

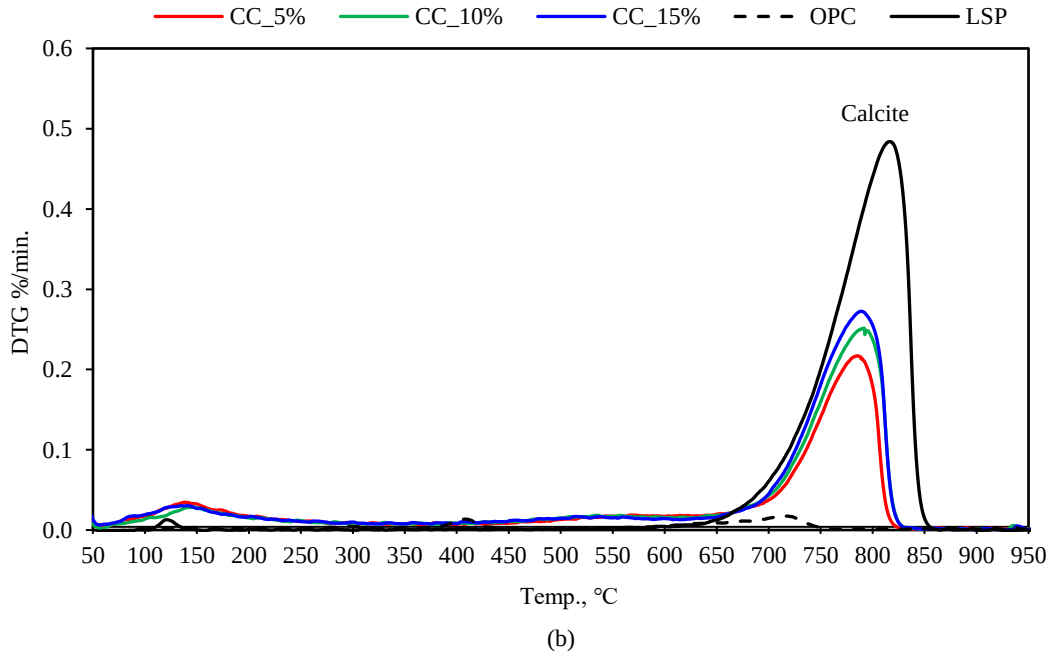
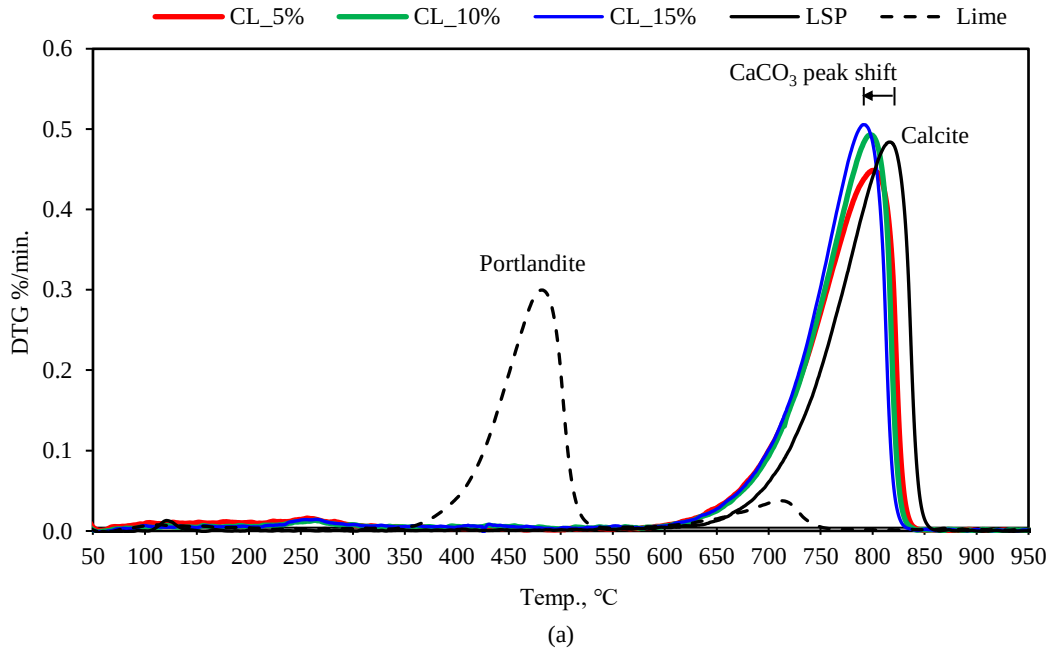


Figure 20: DTG curves of raw/carbonated (a) lime, (b) OPC.

The XRD spectra of fresh and carbonated CRMs are shown in Figure 21, with calcite, portlandite, alite, and belite marked as prominent phases. LSP exhibits prominent calcite peaks, which are a benchmark for identifying carbonate phases in carbonated CRMs. Lime shows a clear portlandite peak, which is absent in CL due to carbonation transforming portlandite into calcite.

For OPC, before carbonation, alite and belite hydrate to form C-S-H and CH [17,149]. During carbonation, these hydrates react with CO_2 , leading to C-S-H decalcification into silica gel and CH conversion to CaCO_3 [97,140–142]. This transformation is evidenced by reduced calcium silicate and increased calcite peaks in the XRD spectrum of CC.

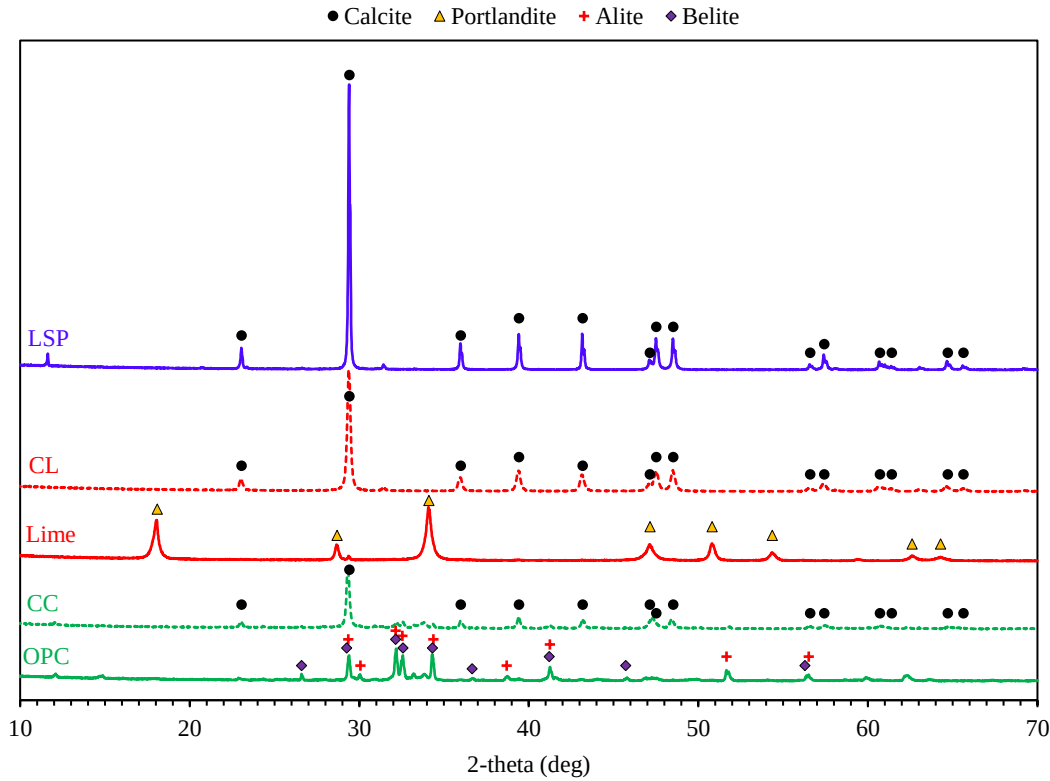


Figure 21: XRD spectra of the fresh and carbonated CRMs.

Figure 22 presents SEM micrographs of fresh and carbonated CRMs. The morphological analysis presented in Figure 22 (a and b) reveals the formation of rhombohedral particles on the surface of OPC following carbonation. This particle shape is characteristic of calcite [264–266], the most thermodynamically stable polymorph of CaCO_3 [267]. Similar morphologies have also been observed in the carbonation of various types of slag [210,268,269]. This observation aligns with DTG profile of CC shown in Figure 20b, where the decomposition temperature range of CaCO_3 in CC matches that typically

observed for calcite in LSP. Conversely, the morphological characteristics of CL, as shown in Figure 22 (c and d), display a mixture of rhombohedral and spherical particles. These shapes correspond to the crystal forms of calcite and vaterite, respectively [264–266,270]. Vaterite, known for its lower stability relative to calcite [267], may account for the observed shift in the CaCO_3 peak in CL's DTG profile compared to LSP.

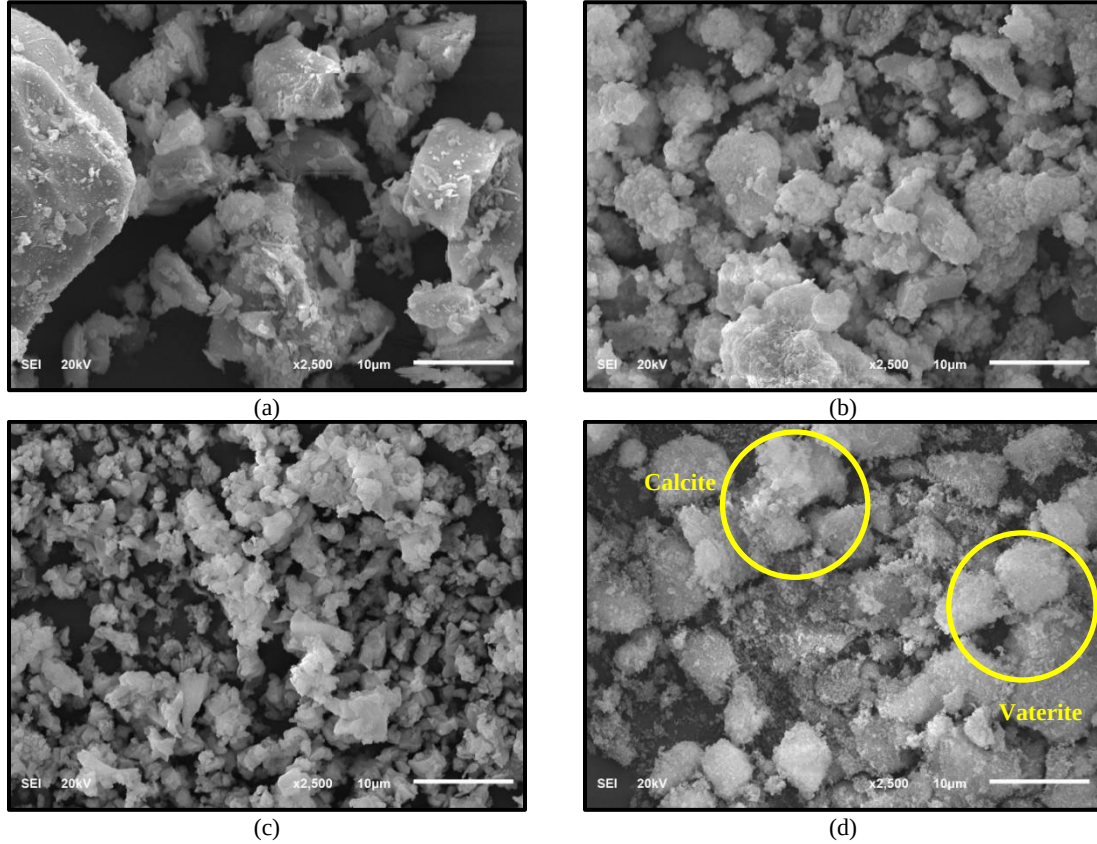


Figure 22: Scanning-electron micrographs (SEMs) of (a) fresh OPC, (b) carbonated OPC, (c) fresh lime, and (d) carbonated lime.

3.4.3 CO_2 uptake capacity and carbonation efficiency

The primary oxides identified through XRF, presented in Table 8, aided to calculate the theoretical CO_2 uptake of OPC, and lime using Steinour's formula (Eq. (30)), while TGA was used to estimate the actual CO_2 uptake during carbonation.

Figure 23 illustrates CO_2 -actual, CO_2 -th and carbonation efficiency of both OPC and lime. Interestingly, both OPC and lime exhibit a comparable theoretical sequestration capacity, hovering around 50%. This similarity is attributed to their equivalent CaO content, as confirmed by the XRF analysis in Table 8. However, the actual CO_2 uptake differed significantly. Lime achieved an actual uptake of 41%, whereas OPC recorded only 22%, leading to a carbonation efficiency of 44% for OPC and 77% for lime.

The reduced efficiency in OPC can be attributed to the formation of C-S-H during hydration, which consumes CH and reduces available lime for carbonation [17,97,140–142,149]. Conversely, pure hydrated lime does not generate competing hydration phases, facilitating greater availability of lime for carbonation. Additionally, the accuracy of TGA measurements might be limited as they are based exclusively on CaCO_3 estimates, potentially missing other carbonate forms that decompose outside the typical CaCO_3 temperature range [271].

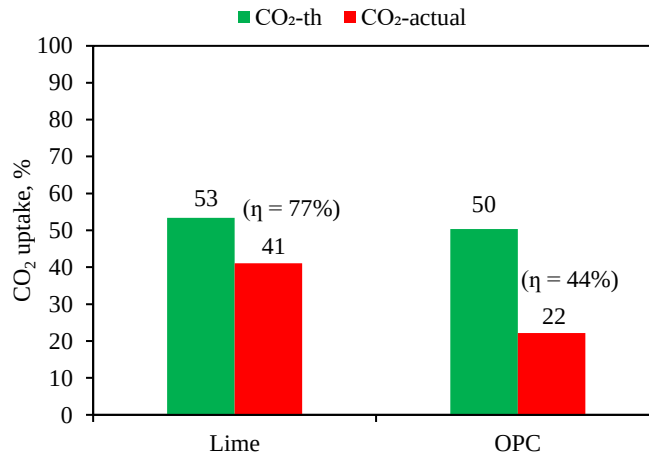


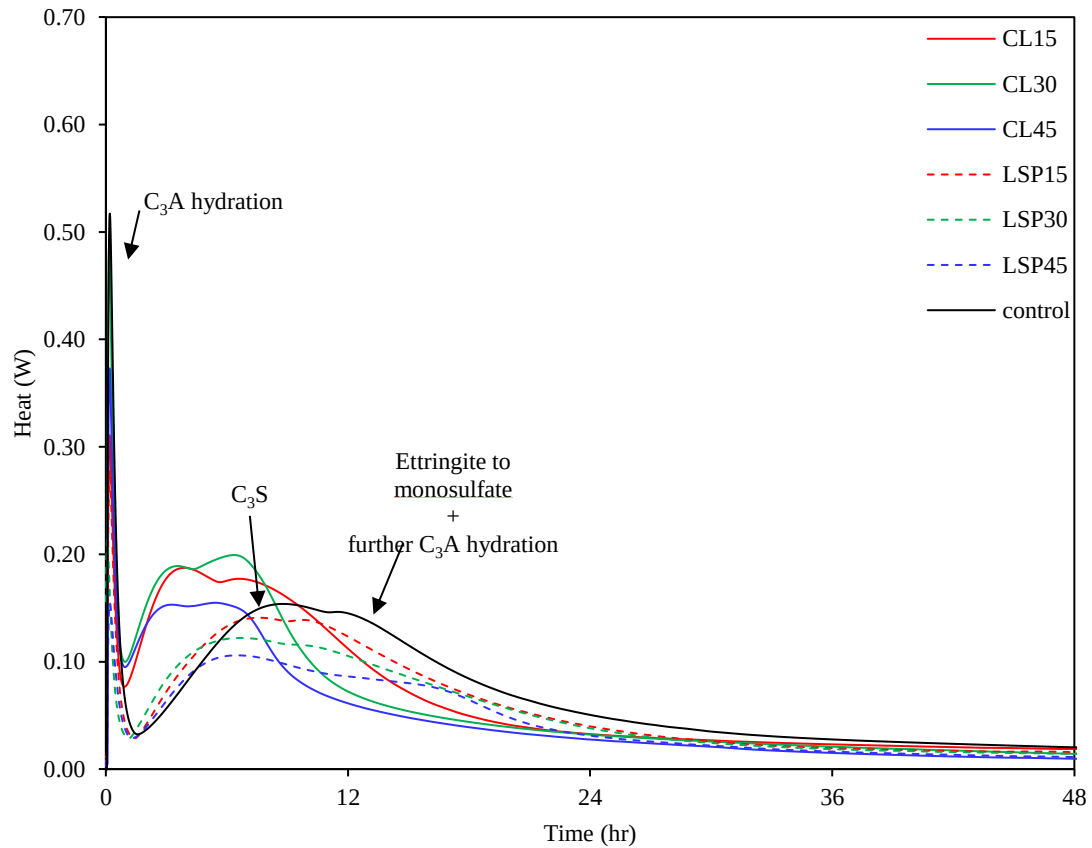
Figure 23: CO₂-actual, CO₂-th and carbonation efficiency of OPC and lime.

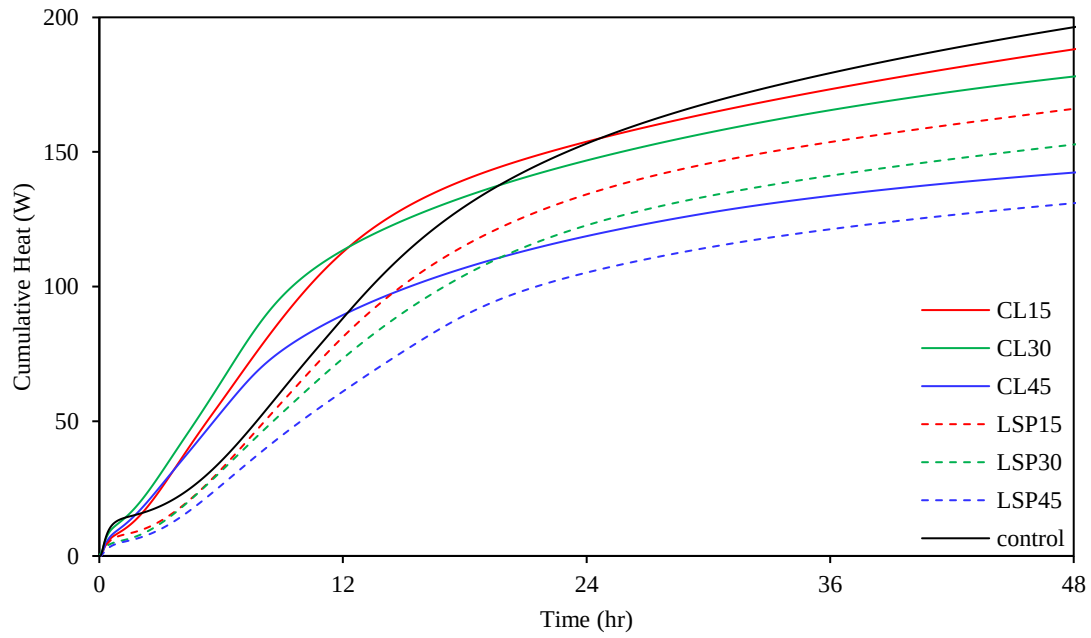
3.4.4 Calorimetry

Figure 24 presents the heat flow of paste mixtures incorporating carbonated CRMs. The heat flow curve is predominantly influenced by the hydration of tricalcium silicate (C_3S) and tricalcium aluminate (C_3A), evidenced by two distinct exothermic peaks at early hydration stages [272,273]. It can be seen that paste mixtures with low replacement levels of CL show that the C_3S peak is amplified and shifted earlier, indicative of accelerated hydration reactions. This increased reactivity can be attributed to the fine particle size of CL, which provides additional surface area, fostering nucleation and growth of hydration products [274–279]. Similar acceleration in early hydration was reported in [278], where early hydration of cement pastes containing 10-20% aqueously carbonated basic oxygen furnace slag was generally faster than in plain cement paste.

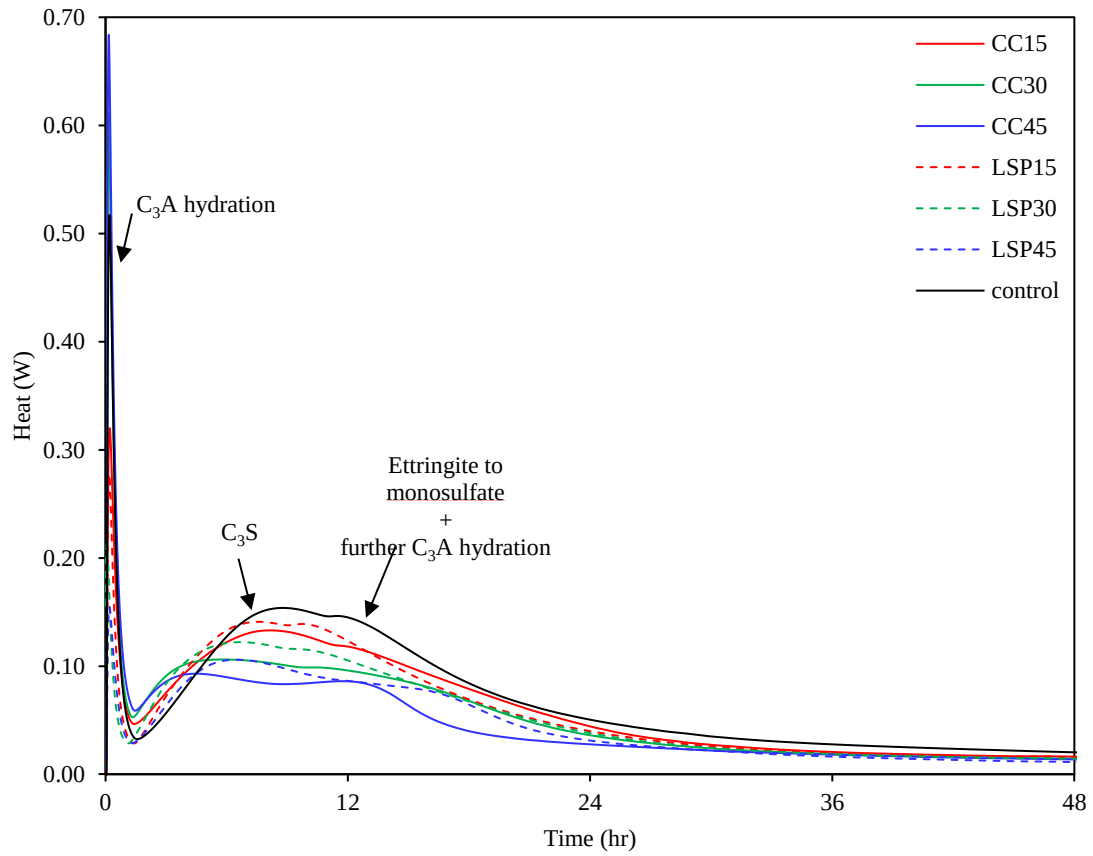
On the other hand, the additions of CC result in lower reactivity, likely attributed to the less reactive nature of CC after carbonation. Additionally, the post-grinding particle size of CC is relatively larger compared to CL, contributing further to its reduced reactivity in the hydration process. Moreover, LSP decreased the reactivity due to the massive size of LSP particles compared to CL and CC particles [274–277]. This reduction in heat evolution with coarse LSP incorporation was also reported in [280].

Additionally, Figure 24 presents the cumulative heat of paste mixtures incorporating carbonated CRMs. As can be seen, the incorporation of carbonated CRMs resulted in a reduction of total heat released during hydration, attributed to the dilution effect due to lower cement clinker content [272,278,281,282]. Comparable trends were observed in [283], where a 30% replacement with aqueously carbonated steel slag led to a 19.3% reduction in cumulative heat. Further analysis in [284] demonstrated a lesser reduction in cumulative heat for ultra-high-performance concrete (UHPC) using carbonated recycled fine aggregate (RFA) compared to UHPC with uncarbonated RFA. In this context, CL mixtures showed lower reductions compared to CC mixtures, while the reductions were most significant in LSP mixtures, reflecting the influence of different fineness.





(a)



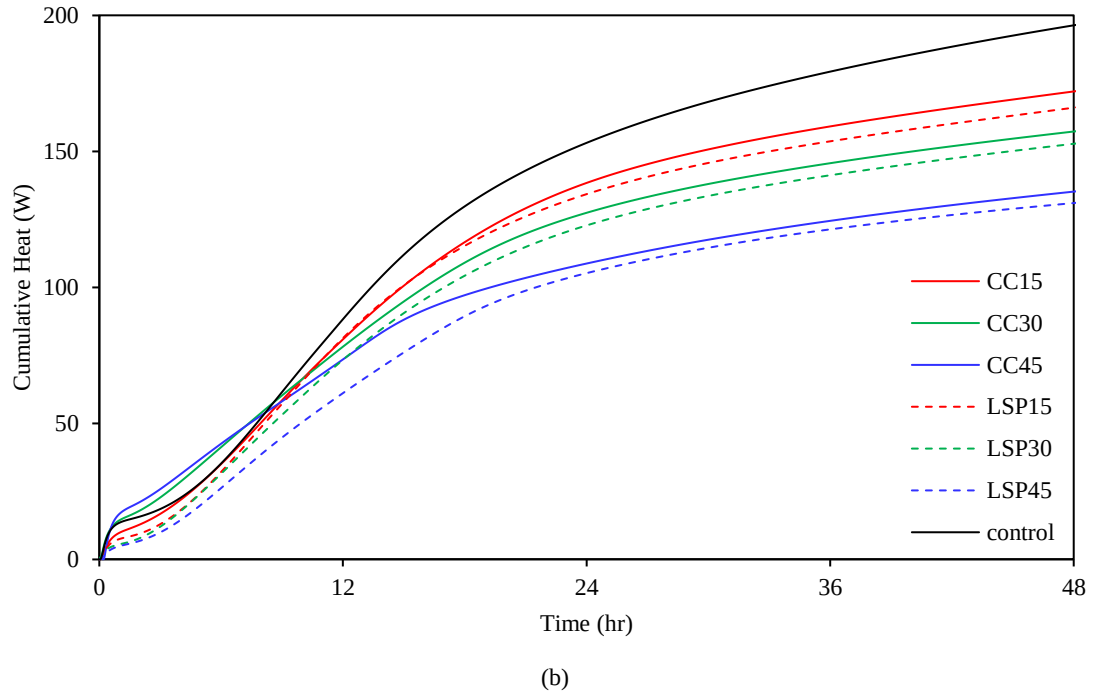


Figure 24: Influence of incorporating (a) CL and (b) CC on the cement paste heat of hydration.

3.4.5 Characteristics of paste mixtures

Figure 25 illustrates the TGA profiles of 28 days-cured cement pastes with 0%, 15%, and 30% replacement by CL, CC, and LSP. With the incorporation of CC and CL, The TGA profiles for 15% replacement reveal that ettringite and CSH peaks are marginally more distinct than the control, indicating a higher formation of hydration products due to the high surface area of CC and CL. As the replacement level rises to 30%, these peaks approach the intensity of the control, reflecting the reduction in cement content and consequent decrease in hydration products. The TGA profiles also underscore the inverse correlation between portlandite and calcite; higher replacement levels with carbonated CRMs yield less portlandite and more calcite. In all instances, LSP indicates a relatively lower quantity of hydration products, highlighting the superior efficacy of carbonated CRMs over natural calcite source of calcite, thus enhancing the sustainability of the cementitious mixtures.

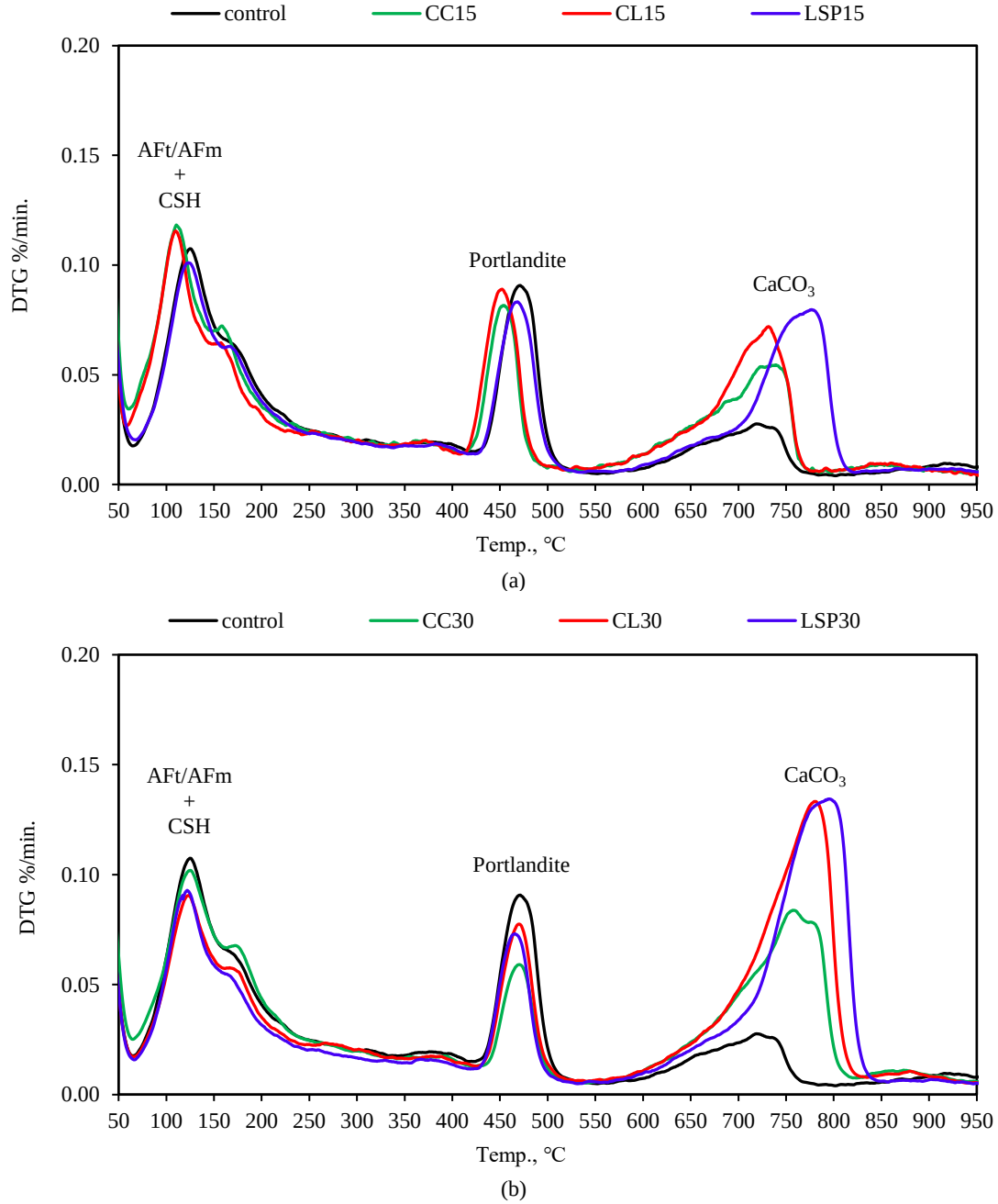


Figure 25: Differential thermogravimetric curve of cement pastes incorporating (a) 10% and (b) 30% carbonated CRMs.

The XRD spectra for 28-day cured cement pastes are presented in Figure 26. The results show that as the replacement level of carbonated CRMs increases, the intensity of ettringite and portlandite peaks decreases while calcite peaks become more pronounced. LSP samples, expectedly, exhibit the highest calcite peaks, correlating with their composition.

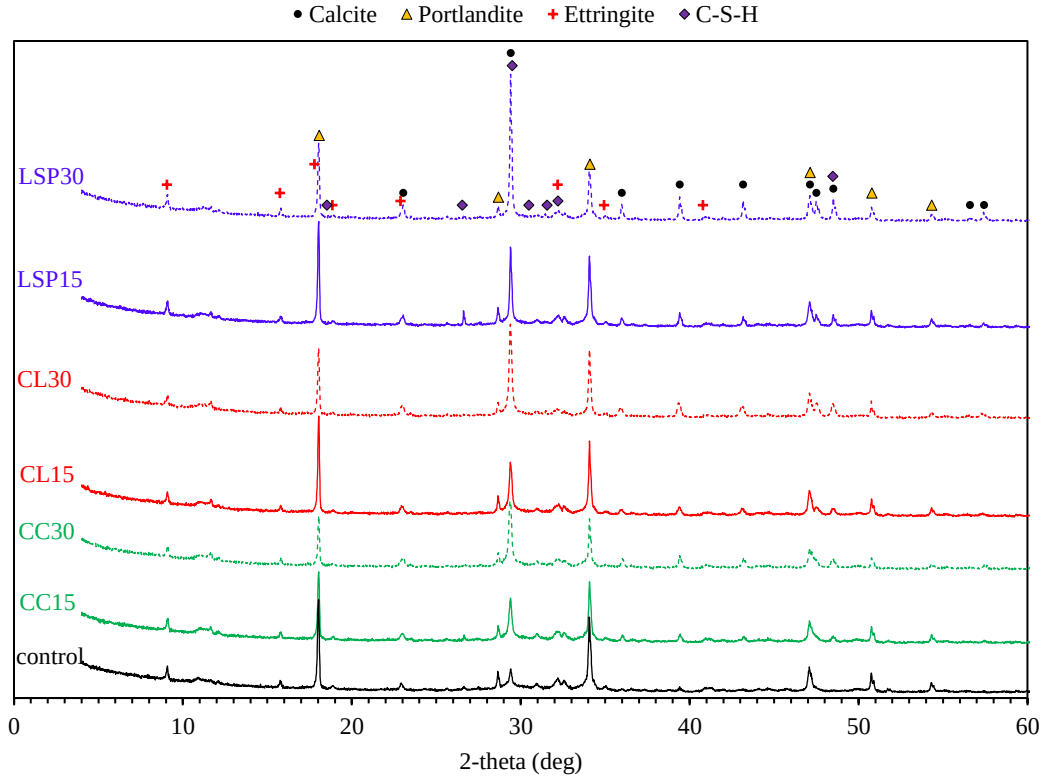
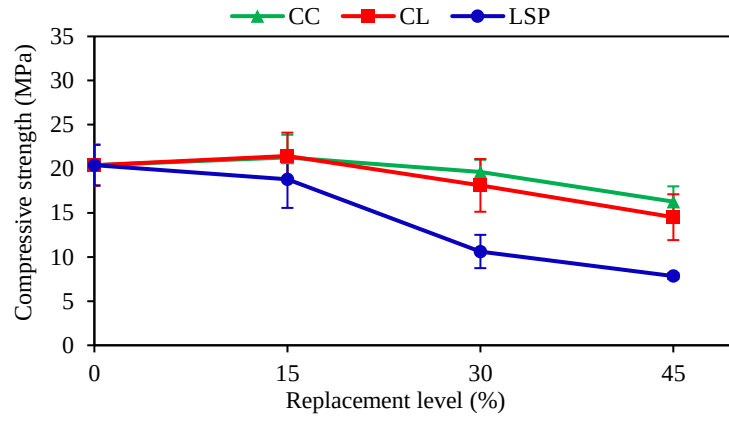
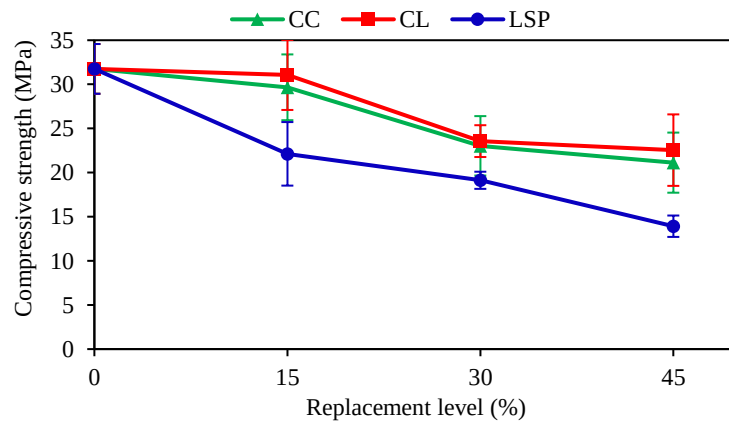


Figure 26: XRD spectra of cement pastes incorporating carbonated CRMs.

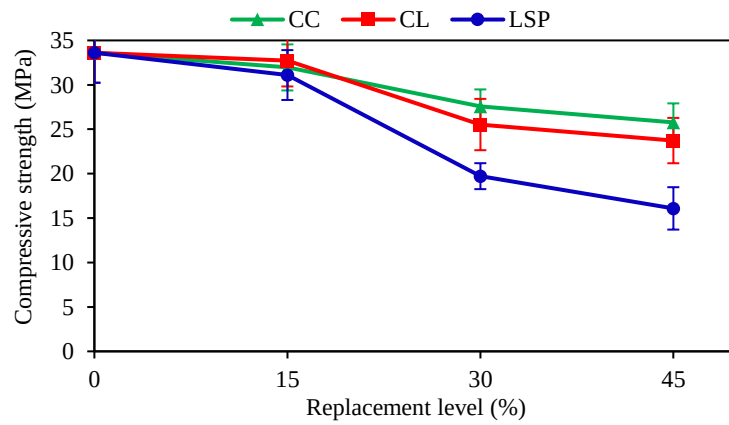
Figure 27 presents the compressive strength development for cement pastes with varying replacement levels of CC, CL, and LSP. At an early age, pastes with a 15% replacement of CL and CC demonstrate slightly enhanced strength, reaching 105% of the control, due to the fine particles of these materials accelerating the hydration reaction and providing additional sites for nucleation, leading to a higher early strength gain. Despite the general decline in compressive strength across all materials as the replacement percentage increases and the curing time extends to 28 days, the incorporation of carbonated CRMs yielded strengths above 20 MPa at all replacement levels. This trend may result from reduced cement content, translating to fewer hydration products that contribute to strength. CC and CL, particularly at lower replacement levels, show potential as substitutes for OPC, offering a sustainable alternative without significantly compromising mechanical properties. Conversely, LSP's larger particle size reduces its reactivity, resulting in a lower quantity of hydration products and leading to diminished strength gains at all ages, more so at higher replacement levels. This indicates that LSP's sustainability benefits might come with a cost in compressive strength.



(a)



(b)



(c)

Figure 27: Compressive strengths of cement pastes incorporating carbonated CRMs at (a) 3 days, (b) 14 days, and (c) 28 days.

3.5 Comparative Analysis of Proposed Carbonation Approach and Existing Technologies

As previously introduced, various carbonation technologies have been developed to enhance CO₂ sequestration in cast-in-place concrete, each employing unique methods and materials. This section revisits these technologies—pre-carbonation and carbonation mixing (gas and liquid CO₂ forms)—and the novel approach developed in this study. Methodologies, effectiveness, and practical applications of these three approaches are compared. Table 10 summarizes the key comparative aspects of these technologies.

3.5.1 Carbonation methodology

Pre-carbonation utilizes a two-stage process where CO₂ is first injected into a slaked lime slurry (made with only 1% concentration), then mixed with other concrete components [18]. Carbonation mixing introduces gaseous CO₂ directly during the concrete mixing process, either as gas [202] or as liquid [203,204]. Conversely, the methodology proposed in this study involves the introduction of CO₂ into a CRM slurry, followed by powdering the carbonated product before being incorporated into OPC-based systems.

3.5.2 Effectiveness in CO₂ sequestration

Carbonation technologies serve two pivotal roles in the concrete industry: they reduce the carbon footprint and enhance the performance of construction materials. A key sustainability indicator for these technologies is the captured CO₂ per megapascal (C-CO₂/MPa), which is used to measure the successfully captured CO₂ relative to strength of OPC-based mixture produced by different carbonation techniques. Although each technology is applied in different phases of OPC-based systems and direct comparisons may be unfair, this analysis provides a general overview of the potential CO₂ absorption for each technology, irrespective of the mixture phase.

For pre-carbonation method, in order to determine the C-CO₂/MPa, it's essential first to calculate the embodied CO₂ per megapascal (E-CO₂/MPa). This analysis assumes the use of the same type of hydrated lime that is used in this study. Table 9 shows the typical E-CO₂ values for OPC and lime. These values represent the majority of E-CO₂ over the life cycle of a structural project [285]. Additionally, the actual CO₂ uptake in Table 9 was used to determine the net E-CO₂. The total net amount of E-CO₂ mortar mixes used in pre-carbonation method were estimated by multiplying the weight of each material (for every cubic meter) by its corresponding E-CO₂, and then summing the results. The E-CO₂/MPa was calculated by dividing the E-CO₂ of each mix by its achieved compressive strength. The C-CO₂/MPa, shown in Figure 28, was then obtained by subtracting E-CO₂/MPa of the

carbonation-treated mortar mix from that of the control mix (prepared without carbonation treatment).

Gas-based carbonation mixing, however, was not considered further in this evaluation due to its failure in maintaining adequate strength. A life cycle assessment of CarbonCure technology indicated that it can absorb an average of 15 kg CO₂/m³ of concrete [286]. Considering a compressive strength of 28.3 MPa achieved by this technology [205], the C-CO₂/MPa was estimated to be approximately 0.53 kg CO₂/MPa, as shown in Figure 28.

Using the same procedure as for pre-carbonation method, the C-CO₂/MPa for paste mixes incorporating 15% carbonated CRMs was calculated. As shown in Figure 28, the proposed methodology exhibits the highest C-CO₂/MPa value among the technologies analyzed. Notably, mixes containing carbonated CC showed a negative C-CO₂/MPa value relative to those with CL. This is attributed to the high E-CO₂ factors of OPC. To mitigate this, utilizing CRMs with relatively lower E-CO₂ factors, such as Class C fly ash (0.008 kg/kg) [285], could reduce the environmental impact when applying this method.

Table 9: Inventory of E-CO₂ [285]

Raw material	OPC	Lime	Sand	Water
E-CO ₂ (kg/kg)	0.912	0.78	0.00493	0.0008
CO ₂ uptake (kg/kg)	0.22	0.41		
Net E-CO ₂ (kg/kg)	0.692	0.37	0.00493	0.0008

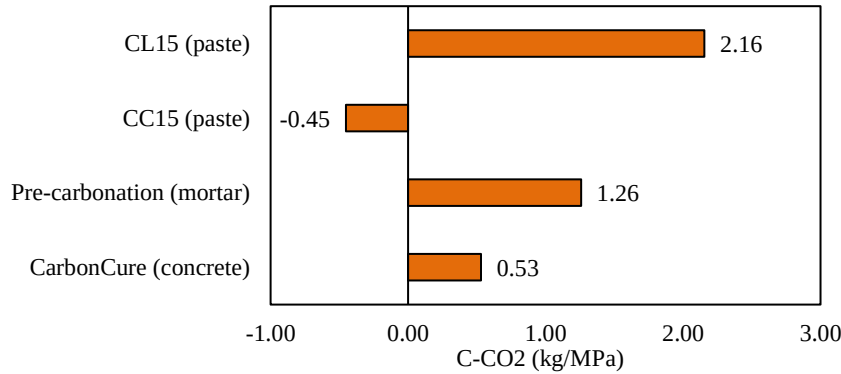


Figure 28: C-CO₂ analysis of recent carbonation technologies.

3.5.3 Impact on strength

The impact of carbonation technologies on the mechanical properties of concrete varies significantly across different methods. Pre-carbonation method achieved a notable increase in the 28-day compressive strength of mortar by 16%. In contrast, gas-based carbonation mixing showed a substantial reduction in compressive strength by 56% [202], which underscores its unsuitability for structural applications. CarbonCure technology (using

liquid CO₂), however, claims to maintain the strength of concrete even with a binder content reduction of up to 7% [205].

The approach proposed in this study showed a 5% reduction in the compressive strength of paste incorporating 15% carbonated CRMs. Although this finding restricts the maximum viable replacement level to less than 15% to avoid compromising strength, it is important to note that pre-carbonation method, with its 1% replacement, and gas carbonation mixing, with significant strength reduction, present limitations in their applicability and sustainability impacts. Additionally, it is critical to highlight that the proposed technology not only may sequester more CO₂ compared to the CarbonCure approach, as mentioned in section 3.5.2, but also promotes the utilization of calcium-rich waste materials. This dual benefit underscores its potential for sustainability, aligning it more closely with environmental goals.

3.5.4 Practicality, scalability, and market potential

Pre-carbonation and carbonation mixing require specific setups for CO₂ injection, therefore, adapting them for on-site concrete mixing is difficult due to equipment challenges and the need to transport and store liquid CO₂ on-site. In Contrast, the technology developed in this study greatly enhances practicality and scalability and holds high market potential by allowing the preparation of dry powders of carbonated CRMs, which can then be bagged, stored, and distributed for use in construction applications in a similar way to other construction materials.

Table 10: Comparative Summary of recent carbonation technologies for cast-in-place concrete (summarized from [18,202,205,206])

Feature	Pre-carbonation	Gas CO ₂ mixing	CarbonCure	Proposed approach
Carbonation methodology	Pre-carbonating lime slurry (1% concentration).	CO ₂ injection during mixing.	CO ₂ injection during batching.	CO ₂ injection into CRM slurry, followed by drying and powdering.
CO ₂ state	Gaseous.	Gaseous.	Liquid.	Gaseous.
Tested phase	Mortar.	Paste.	Concrete.	Concrete.
28-Days strength increase	+16%.	-56%.	-7% binder. 100% strength.	-5% at 15% replacement
Adaptability for on-site use	Requires CO ₂ setup.	Requires CO ₂ setup.	Requires liquid CO ₂ setup.	Easy transport and use of dry powder.

3.6 Conclusions

The study presented evaluates recent carbonation techniques in concrete and addresses their key limitations by introducing an innovative off-site carbonation approach designed especially for on-site concrete mixing applications. Selecting hydrated lime and ordinary Portland cement (OPC) as calcium-rich materials (CRMs), the proposed approach involves injecting CO₂ into slurries of these CRMs, powdering the carbonated products, and then incorporating them into concrete mixtures. Limestone powder (LSP) was used to serve as a mineral carbonate benchmark to study the behaviors of the carbonated products – carbonated lime (CL) and carbonated cement (CC). The production and analysis of carbonated CRMs as well as the effects of incorporating carbonated CRMs on the hydration kinetics, compressive strength, and microstructure of cement paste are discussed. The following conclusions can be drawn from the outcomes of this study:

Microstructure analysis confirms calcite as the dominant carbonation product of the CRMs. This is evidenced by DTG peaks and increased calcite peaks in XRD spectra. Changes in surface morphology observed in SEM micrographs revealed that carbonation of CRMs results in the formation calcite particles in OPC. In contrast, carbonation of lime shows both calcite and the less stable vaterite, as indicated by the earlier thermal decomposition of CaCO₃ in DTG curves.

Isothermal calorimetry results showed that incorporation carbonated CRMs alters hydration kinetics and heat evolution in paste mixtures. CL enhances reactivity due to its fine particle size, followed by CC, while LSP exhibited the lowest reactivity owing to larger particle sizes.

Compressive strength results highlighted that at 15% cement replacement, CL and CC enhanced early-age compressive strength by up to 5% and maintained more than 95% of the control mixture after 28 days. However, strengths generally decreased at the high replacement levels. Nonetheless, CL and CC maintained above 20 MPa strength in contrast to LSP mixtures that fell below 20 MPa strength.

The stated impacts of carbonated CRMs on strength were supported by TGA and XRD analyses. At a 15% cement replacement level, CC and CL enhanced the hydration products as indicated by more pronounced DTG and XRD peaks, whereas declines in those peaks were observed at 30% replacement.

Comparative analysis of carbonation technologies for concrete revealed varied features. Pre-carbonation and gas-based CO₂ mixing showed limited benefits due to low replacement levels and reduced strength, respectively. Solid-based CO₂ mixing maintained strength with moderate CO₂ absorption. The innovative approach using carbonated CRMs

at low replacement excels in CO₂ sequestration and practical application and effectively maintained adequate strength.

3.7 Recommendations

The proposed method effectively utilizes CO₂ for environmental benefits. While pure CO₂ was used in this study, practical applications could leverage flue gas as a more cost-effective CO₂ source. OPC and lime were used in this work as a base to try out the proposed carbon utilization method in cementitious composites. Calcium-rich wastes, such as Class C fly ash and slag, can be a better sustainable alternative to replace OPC and lime in real-world applications of this carbonation technique. Waste cement recovered from plant and truck washouts in ready-mix plants is particularly close to the 1-hour hydrated OPC utilized in this study. Therefore, the findings reported herein for the carbonated OPC offer valuable information for industrial upcycling of waste cements into a carbon-trapping construction material that benefits the environment. Since this study primarily focused on the incorporation of carbonated CRMs in blended paste mixtures, further investigation is necessary to understand their performance in mortar and concrete mixtures.

3.8 Acknowledgements

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3.9 CRediT Author Contribution Statement

Omer Ahmed: Formal analysis, Investigation, Data Curation, Writing – Original Draft, Writing – Review & Editing, Visualization. **Saheed Kolawole Adekunle:** Conceptualization, Methodology, Validation, Resources, Writing – Review & Editing, Visualization, Supervision, Project administration, Funding acquisition. **Shamsad Ahmad:** Conceptualization, Methodology, Validation, Writing – Review & Editing, Supervision. **Mohammed Ali Alosta:** Validation. **Ahmed Abd El Fattah:** Validation. **Mesfer Mohammad Al-Zahrani:** Validation.

CHAPTER 4

EFFECT OF CARBONATED CALCIUM-RICH MATERIALS ON MECHANICAL AND DURABILITY PROPERTIES OF OPC MIXTURES

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4.1 Abstract

Accelerated carbonation is getting recognized as an effective strategy for reducing the concrete industry's carbon footprint. Yet, the required conditions to facilitate concrete accelerated carbonation restricts its use to precast concrete applications. Recent studies have explored the possibility of incorporating CO₂ directly into fresh concrete, but these methods feature inherent complexity for on-site concrete mixing. Addressing these challenges, this study introduces a novel approach to introducing CO₂ directly into fresh concrete in three steps. Using hydrated lime and ordinary Portland cement (OPC) as calcium-rich materials (CRMs), the method involves injecting CO₂ into CRM slurries, converting these into powdered form, and finally blending them into mortar/concrete mixtures. Compressive strength results revealed that at a 10% OPC replacement level, mortar mixtures with carbonated cement (CC) and carbonated lime (CL) show better performance than those with limestone powder (LSP), retaining 95% of the control's strength and elastic modulus, while LSP mixture maintained only 87%. Tensile strength, drying shrinkage, and water absorption were better in CC and CL than in LSP mixtures. CC mixtures exhibited the most insignificant rise in chloride migration coefficient even at maximum replacement level, exceeding the control mixture's value by only 28%. A 20% replacement level was identified as optimal for balancing environmental benefits with strength. The blended concrete mixtures produced at 20% replacement demonstrated compressive strength, elastic modulus, and water absorption comparable to the control mixture. Sustainability analysis of mortar and concrete mixtures suggested that incorporating carbonated CRMs in mortar and concrete can effectively reduce the embodied CO₂.

Keywords: carbon dioxide, mineral carbonation, calcite, cement, concrete.

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4.2 Introduction

The cement and concrete sectors are currently recognized as major sources of global carbon dioxide (CO₂) emissions, contributing to nearly 8.0% of worldwide emissions [248,249]. In 2019, the market value of these products stood at \$370.1 billion and is expected to witness a significant expansion. Predictions indicate a 12% compound annual growth rate from 2021, with global cement output reaching 5 billion tons by 2030 [248]. Consequently, it is projected that emissions from cement and concrete production could account for more than 10% of the global CO₂ emissions from 2030 to 2050 [248].

The production of ordinary Portland cement (OPC) is known to emit CO₂, which is subsequently reabsorbed by concrete products through a gradual and natural chemical process. Yet, this reabsorption process is notably slow, potentially requiring several centuries to counterbalance the initially released CO₂ [14,15]. This inefficiency has amplified the urgency to align the substantial worldwide demand for cement-based materials with initiatives to limit greenhouse gas emissions. It is crucial for the concrete industry to adopt and implement effective methods to foster sustainable growth [248,249]. An established approach is the replacement of cement clinker with alternative cementitious materials [250–252], given that clinker production accounts for approximately 85% of the total CO₂ emissions from concrete [253]. Nonetheless, relying only on this method is inadequate to meet the global objectives for CO₂ emission reduction.

In the pursuit of reducing CO₂ emissions, approaches such as carbon capture, utilization, and storage (CCUS) are also being explored. CCUS encompasses the process of capturing CO₂ from industrial sources, followed by its utilization or sequestration in different forms [254]. One approach within this strategy is accelerated carbonation curing (ACC), where early-age concrete is exposed to highly concentrated CO₂. This exposure facilitates a chemical reaction between the CO₂ and the concrete to form solid calcium carbonates (CaCO₃). Such a process not only strengthens the concrete [154,159,162–168] but also limits the water absorption [167] and chloride migration [167,173] in concrete. Additionally, mineral carbonation is another CCUS technique, focusing on the stable sequestration of CO₂ in carbonate forms such as CaCO₃ and MgCO₃. This technique advances the natural carbonation of calcium and magnesium-rich materials, resulting in the permanent storage of CO₂. Mineral carbonation has been primarily implemented using alkaline waste materials, such as steel slag [53], recycled concrete aggregates [59–61,73–

75], and fly ash [56–58,71,72], instead of natural resources to preserve nature and minimize energy usage in material grinding and extraction. These waste materials are selected for their rapid reaction kinetics, low energy demands, and high efficiency in carbonate formation compared to natural minerals [48,49]. However, the application of these carbonated materials as ingredients in Portland cement concrete remains relatively unexplored.

A study attempted to introduce mineral sequestration into OPC mortar by pre-carbonating slaked lime slurry and mixing the carbonated material with other components [18]. The findings revealed that incorporating 1% carbonated lime enhanced the compressive and flexural strength of the mortar by 16% and 5%, respectively, and modestly decreased the 28-day drying shrinkage. While these outcomes are promising, additional investigations are required to gather more data on the impact of increased proportions of carbonated lime in cementitious composites and its environmental benefits when used as a substitute for cement. Moreover, the feasibility of this process and its application for on-site settings presents significant challenges that need to be addressed.

Similarly, previous studies have introduced CO₂ during the concrete mixing stage, a method referred to as carbonation mixing [197,198,202]. This technique involves continuously injecting CO₂ gas at a steady rate into a concrete mixer. However, it has been observed that this process may significantly decrease the strength of the cement paste [202]. In an alternative strategy, liquid CO₂ has been used instead of gaseous CO₂ [203,204], which has been commercialized as CarbonCure, a retrofit technology for ready-mix concrete plants. This technology involves the precise injection of liquid CO₂ during the batching stage of ready-mix concrete. Upon contact with fresh concrete, liquid CO₂ changes into a mixed gas-solid state, similar to dry ice flakes, due to a significant pressure drop. CO₂ rapidly reacts with calcium ions to form CaCO₃, which is then permanently embedded within the concrete matrix. The proponents claim that this technology restores the compressive strength of concrete made with 7% binder reduction [205]. Despite the apparent advantages of carbonation mixing, implementing it directly on construction sites presents difficulties due to logistical and equipment challenges. Thus, a revised approach is necessary to address these obstacles.

To address these research gaps, this study proposes preparing dry powders of carbonated CRMs, which can be packaged, stored, and utilized similarly to conventional construction materials. The process considered the use of dry carbonated forms of two CRMs, introducing mineral sequestration into the fresh OPC-based mixtures in three stages. Initially, CO₂ is introduced into a CRM slurry, and the carbonated product is then filtered, dried, and ground into powder. The final step includes incorporating the final product in concrete. This approach is ideal for on-site applications since the carbonation process is conducted off-site, enabling the powdered product to be stored and used as a cement substitute.

Aiming to explore this method, this paper reports a study conducted on the production of two different carbonated CRMs and the effect of their incorporation at various levels on the mechanical and durability properties of mortar and concrete. Simultaneously, the study also assesses CO₂ uptake capacity and carbonation efficiency of these carbonated materials and performed a comprehensive sustainability analysis on the produced mortar/concrete mixtures to evaluate the environmental impact of this method.

4.3 Experimental Program

4.3.1 Materials

Two primary materials were considered in this study as calcium-rich materials (CRMs): industrial hydrated lime and ordinary Portland cement (OPC), complying with ASTM C 150. Industrial CO₂ gas with 99.9% purity was used for carrying out the carbonation process. Limestone powder (LSP) was used as a natural calcium carbonate (CaCO₃) benchmark to study the effects of incorporating the chemically precipitated CaCO₃ resulting from the carbonation of CRMs. As obtained by X-ray fluorescence (XRF) analysis, the chemical compositions of primary study materials are presented in Table 11.

Dune sand with a specific gravity of 2.54 and water absorption of 0.7% was used as fine aggregate. Crushed stone with a maximum size of 9.5 mm, specific gravity of 2.6, and water absorption of 0.64% was used as coarse aggregate.

Table 11: Chemical composition (m/m %) of lime, OPC, and LSP.

Oxide	Lime	OPC	LSP
CaO	68.01	64.64	32.66
SiO ₂	0.29	17.30	7.27
Al ₂ O ₃	0.64	4.00	1.77
Fe ₂ O ₃	0.13	3.19	1.21
SO ₃	0.09	1.65	0.15
K ₂ O	0.07	1.04	0.82
P ₂ O ₅	0.55	0.67	0.42
TiO ₂	0.02	0.24	0.16

4.3.2 Production of carbonated CRMs

Three different slurries were prepared by mixing samples of each CRM with 500 ml deionized water in a 1-liter flask at a concentration of 15% (by weight of water). Each CRM slurry was left to hydrate for 1 hour, after which the hydrated slurry was carbonated by directly injecting CO₂ gas through the slurry at a flow rate of 6 L/min. A magnetic stirrer was employed to agitate the slurry during carbonation. To ensure a high degree of carbonation at a minimum duration, the pH level of the carbonating slurry was continually monitored at 5-minute intervals. The carbonation process was terminated when the pH attained a steady state.

During this process, CO_2 combines with water to form carbonic acid (H_2CO_3), which dissociates into positively charged hydrogen ions (H^+) and negatively charged carbonate ions (CO_3^{2-}). These ions react with calcium ions (Ca^{2+}) released from CRM in solution [41,62–64], resulting in the precipitation of CaCO_3 as nano to sub-micrometer particles.

The carbonated CRM was recovered from the slurry via vacuum filtration (using a Grade 3 filter paper with $5\ \mu\text{m}$ particle retention) and dried in an oven at 40°C until a stable weight was attained. Finally, the resulting carbonate was pulverized using a low-power blender to disperse the clumped dry mass into a powdery material. The whole process of preparing a powdery carbonated CRM is depicted in Figure 29.

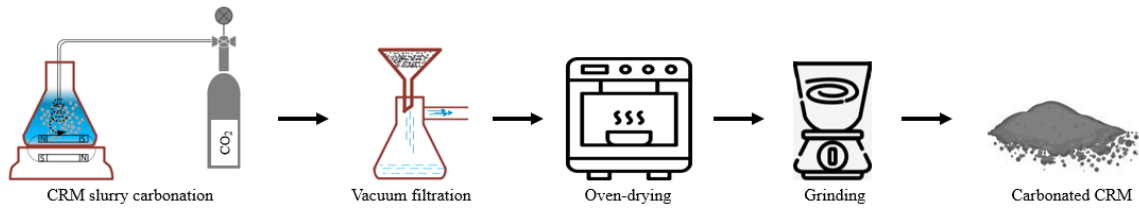


Figure 29: Production of carbonated CRMs.

4.3.3 Material characterization

The particle size distribution (PSD) of the raw and pulverized carbonated CRMs was obtained using an Anton Paar particle size analyzer in a wet medium of ethanol. Water was intentionally avoided as a dispersant in order to prevent any potential chemical reactions with the CRMs that could corrupt the PSD results. The specific gravities of raw and carbonated CRMs were determined using a 50-ml pycnometer filled with ethanol. Thermogravimetric analysis (TGA) of fresh and carbonated CRMs was carried out by heating materials from 30°C to 1000°C at 10°C per min using Perkin Elmer STA 6000 to quantify their CaCO_3 contents.

4.3.4 CO_2 uptake capacity and carbonation efficiency

The carbonation of cement products serves two primary objectives in the concrete industry: reduction of carbon footprint and enhancement of the construction materials' performance. These objectives are closely linked to essential factors: the capacities of carbonatable materials to absorb CO_2 and carbonation efficiency (or degree of carbonation) [93,94,98,99].

In this study, the theoretical maximum CO_2 uptake ($\text{CO}_{2\text{-th}}$) was calculated using Steinour's formula, presented in Eq. (32), based on the chemical composition of CRMs in terms of weight percentage, as reported in previous studies [255–258]. The actual CO_2 uptake ($\text{CO}_{2\text{-actual}}$) was determined using TGA.

$$\text{CO}_{2\text{-th}} = 0.785 (\text{CaO} - 0.56 \text{CaCO}_3 - 0.7 \text{SO}_3) + 1.091 \text{MgO} + 0.71 \text{Na}_2\text{O} + 0.468 \text{K}_2\text{O} \quad (32)$$

Eq. (33) was used to calculate the carbonation efficiency (η), representing the amount of CO_2 successfully sequestered by a material compared to its theoretical sequestration capacity.

$$\eta (\%) = \frac{\text{CO}_{2\text{-actual}}}{\text{CO}_{2\text{-th}}} * 100 \quad (33)$$

4.3.5 Preparation and characterization of OPC-substituted mortars

Table 12 shows the mix proportions of mortar mixtures considered in this study. Labels CL, CC, and LSP denote carbonated lime, carbonated OPC, and LSP, respectively. The number after the CRM type indicator stands for the percentage of cement replaced by CRM in the mixture. Mixtures prepared with binder: sand: water ratio of 1: 2.75: 0.5.

Table 12: Mix proportion for the produced cement mortars (kg/m³)

Mixture	OPC	CRM	Water	Sand
Control	539.03	0	1482.3	288.06
CC10	485.12	53.903	1482.3	288.06
CC20	431.22	107.81	1482.3	288.06
CC30	377.32	161.71	1482.3	288.06
CL10	485.12	53.903	1482.3	288.06
CL20	431.22	107.81	1482.3	288.06
CL30	377.32	161.71	1482.3	288.06
LSP10	485.12	53.903	1482.3	288.06
LSP20	431.22	107.81	1482.3	288.06
LSP30	377.32	161.71	1482.3	288.06

Compressive strength

Compressive strength was carried out in accordance with ASTM C109 [287]. Cubical cement mortar samples (50 × 50 × 50 mm) were manufactured using the mix proportions shown in Table 12. The samples were demolded after 24 hours of casting and cured under damp burlap at 23°C for 3, 7, and 28 days.

Elastic modulus

Elastic modulus was conducted according to ASTM C469 [288]. Cylindrical cement mortar samples (75 × 100 mm) were manufactured using the mix proportions shown in Table 12. The samples were demolded after 24 hours of casting and cured under damp burlap at 23°C for 28 days.

Splitting tensile strength

Splitting tensile strength was carried out following ASTM C 496 [289]. Cylindrical cement mortar samples (75×100 mm) were manufactured using the mix proportions shown in Table 12. The samples were demolded after 24 hours of casting and cured under damp burlap at 23°C for 28 days.

Drying shrinkage

Drying shrinkage was assessed following the guidelines set by ASTM C 596 [290]. Using the mix proportions shown in Table 12, $25 \times 25 \times 285$ mm prismatic cement mortar samples were manufactured. After being removed from the molds at 24 hours, the samples underwent a curing process in lime-saturated water. Then samples were removed from this solution after 72 hours. A digital comparator was used for the initial size measurements. Subsequently, the samples were placed in a controlled environment oven, which was maintained at 23°C and 55% relative humidity. Measurements for drying shrinkage were taken at 7, 14, 21, and 28 days.

Water absorption

Water absorption was assessed in accordance with ASTM C1403 [291]. Using the mix proportion shown in Table 12, $50 \times 50 \times 50$ mm cubic cement mortar samples were manufactured. The samples were demolded after 24 hours of casting and cured under damp burlap at 23°C for 28 days.

Chloride migration

Chloride migration was assessed in accordance with NT BUILT 492 [292]. Using the mix proportion shown in Table 12, 100×200 mm cylindrical cement mortar samples were manufactured. The samples were demolded after 24 hours of casting and cured under damp burlap at 23°C for 28 days.

4.3.6 Preparation and characterization of OPC-substituted concrete

Table 13 and Table 14 show concrete mixtures' parameters and quantities used in this study. Cylindrical concrete samples (100×200 mm) were produced, demolded after 24 hours of casting, and cured under damp burlap at 23°C for 28 days. The samples were tested for compressive strength (ASTM C39 [293]), elastic modulus (ASTM C469 [288]) and water absorption (ASTM C642 [294]).

Table 13: Concrete mix parameters

Parameter	Method/value
Mix design method	Absolute volume method
Mix	Control, CC20, CL20, LSP20
Replacement level	0, 20%
FA/TA	0.45
OPC content	380 kg/m ³
w/b ratio	0.425

Table 14: Concrete mix proportions

Material	Quantity (kg/m ³)			
	Control	CC20	CL20	LSP20
OPC	380	304	304	304
CRM	0	76	76	76
Water	162	162	162	162
FA	814	806	803	804
CA	995	985	982	983

4.3.7 Environmental impact assessment

Embodied CO₂ (E-CO₂) and energy (EE) are the most important sustainability indicators for concrete production. In this study, the impact of this carbonation method on E-CO₂ and EE was evaluated in order to investigate its potential for reducing the environmental impact of concrete production.

4.4 Results and Discussions

4.4.1 Characteristics of carbonated CRMs

Particle size distribution (PSD) and specific gravity (SG)

Figure 31 and Figure 32a show the PSD and mean particle size of the fresh and carbonated CRMs, respectively. The data clearly indicates that carbonated CRMs exhibit a finer PSD when compared to their fresh counterparts, resulting in the mean size reduction of 29.4 and 14% in carbonated OPC and lime, respectively. This observation can be primarily ascribed to the grinding process employed to declump the carbonated materials. Although its primary purpose was to break the dried material clumps of the carbonated CRMs into powder, the grinding process is expected to reduce the materials' particle sizes. The carbonated CRM particles are composed of loose outer and inner CaCO₃ particles that easily break down into finer particles under the mechanical energy imparted by the blender, as depicted in Figure 30. Consequently, the increased surface area of the finer carbonated CRMs enhances the reactivity of the materials.

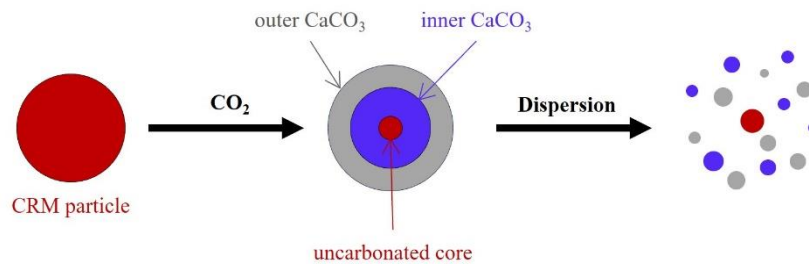


Figure 30: Morphology transformation mechanism of CRM particle.

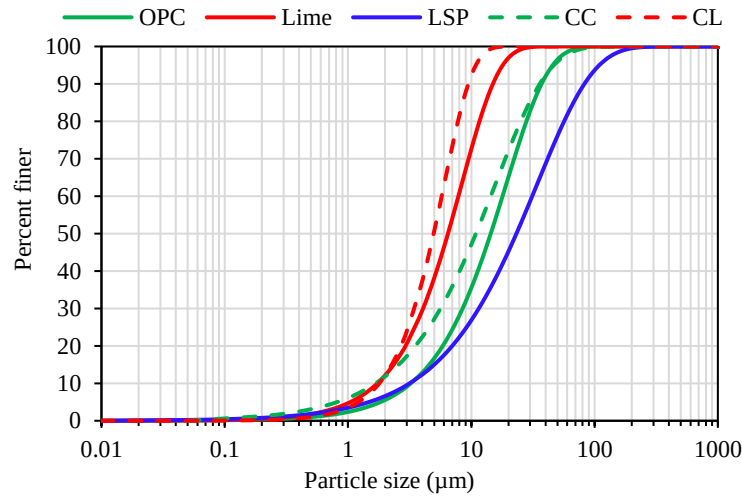


Figure 31: Particle size distribution of the raw and carbonated CRMs.

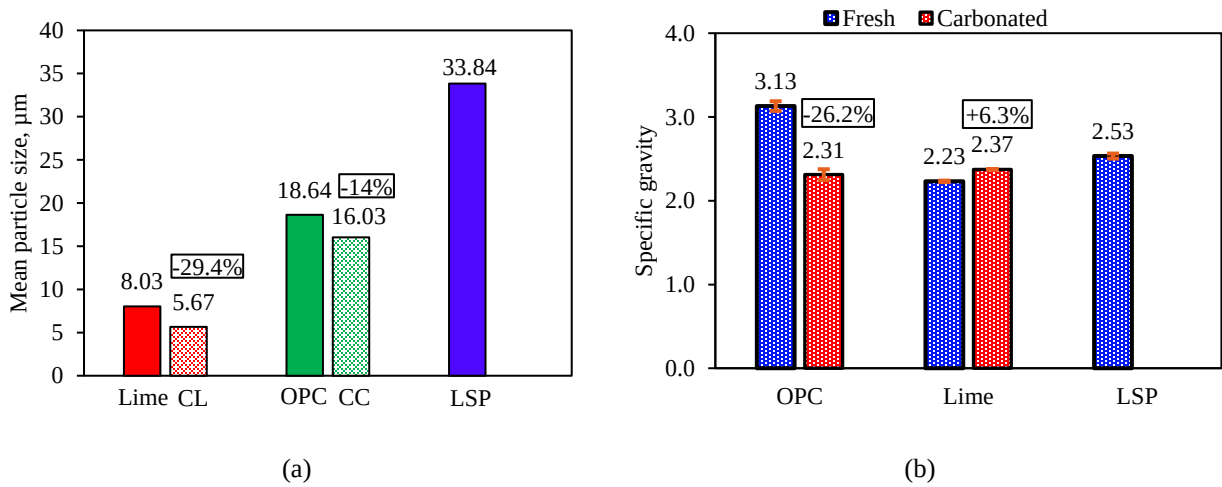


Figure 32: Effect of carbonation on (a) mean particle sizes and (b) specific gravity of CRMs.

Figure 31 shows the specific gravity (SG) of raw and carbonated CRMs. The data distinctly shows that CC exhibited a reduction in SG by 26.2% relative to its uncarbonated counterpart. This decrease is attributed to the formation of CaCO_3 , which has a lower density than raw OPC minerals [260], effectively reducing the overall SG. On the other hand, the SG of CL increased by 6.3% compared to raw calcium hydroxide. This increase is attributed to the higher density of CaCO_3 formed during carbonation [260,261], which elevates the overall SG.

Thermogravimetric Analysis

Figure 33 presents the DTG profiles of the fresh and carbonated CRMs, describing the transformations accompanying the thermal decomposition of carbonated lime and OPC phases. The results reveal a pronounced DTG peak in the temperature range of 400°C to 500 °C on the hydrated lime curve, signifying the thermal decomposition of portlandite. The mineral carbonate benchmark material LSP is principally composed of calcite, the most thermally stable CaCO_3 polymorph [262,263]. The LSP decomposition profile exhibits its characteristic high thermal stability indicated by higher decomposition temperatures, between 700 °C and 800 °C.

Comparing the DTG profiles of the CL with that of LSP, it can be noted that the CaCO_3 precipitated in CL exhibits thermal stabilities close to the calcite in LSP, albeit with a slight backward shift in peak temperature (roughly indicating degrees of crystallinity). It may then be inferred that calcite is the dominant CaCO_3 polymorph precipitated in CL.

In contrast, CC exhibits a less intense peak in the same temperature range, implying a lower CaCO_3 content. This observation is consistent with the expected lower free Ca^{2+} concentration in 1-hr hydrated OPC, as free Ca^{2+} from lime and C-S-H are the primary reacting species in the carbonation reaction leading to CaCO_3 formation. Further, the closeness of the CC's decomposition profile to LSP's also suggests calcite as the dominant carbonation product in CC.

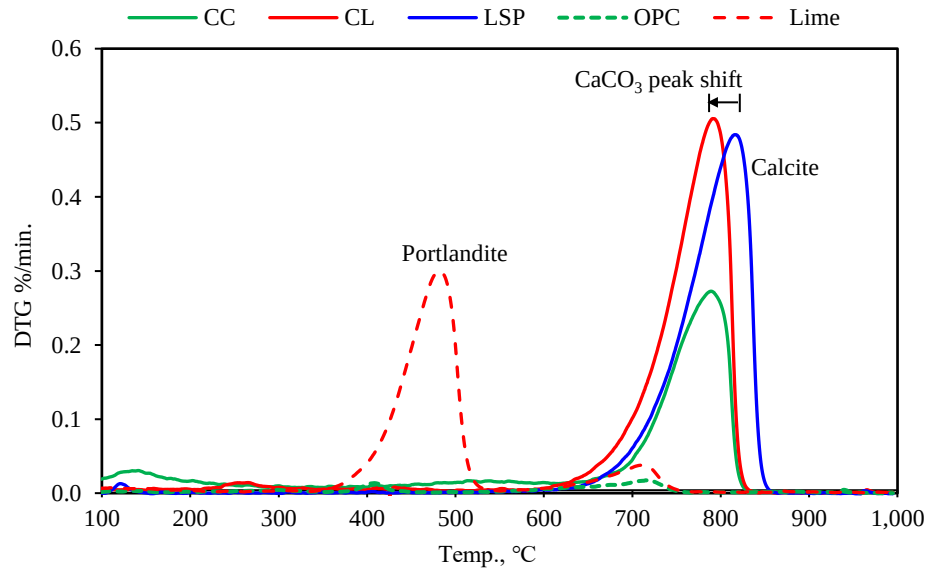


Figure 33: DTG curves of raw/carbonated CRMs.

4.4.2 CO₂ uptake capacity and carbonation efficiency

The primary oxides identified through XRF, presented in Table 8, aided to calculate the theoretical CO₂ uptake of OPC, and lime using Steinour's formula (Eq. (32)), while TGA was used to estimate the actual CO₂ uptake during carbonation.

Figure 34 illustrates CO₂-actual, CO₂-th and carbonation efficiency of both OPC and lime. Interestingly, both OPC and lime exhibit a comparable theoretical sequestration capacity, hovering around 50%. This similarity is attributed to their equivalent CaO content, as confirmed by the XRF analysis in Table 8. However, the actual CO₂ uptake differed significantly. Lime achieved an actual uptake of 41%, whereas OPC recorded only 22%, leading to a carbonation efficiency of 44% for OPC and 77% for lime.

The reduced efficiency in OPC can be attributed to the formation of C-S-H during hydration, which consumes CH and reduces available lime for carbonation [17,97,140–142,149]. Conversely, pure hydrated lime does not generate competing hydration phases, facilitating greater availability of lime for carbonation. Additionally, the accuracy of TGA measurements might be limited as they are based exclusively on CaCO₃ estimates, potentially missing other carbonate forms that decompose outside the typical CaCO₃ temperature range [271].

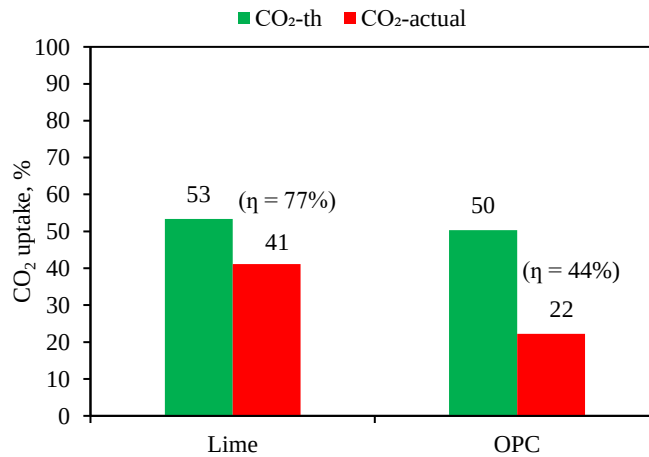
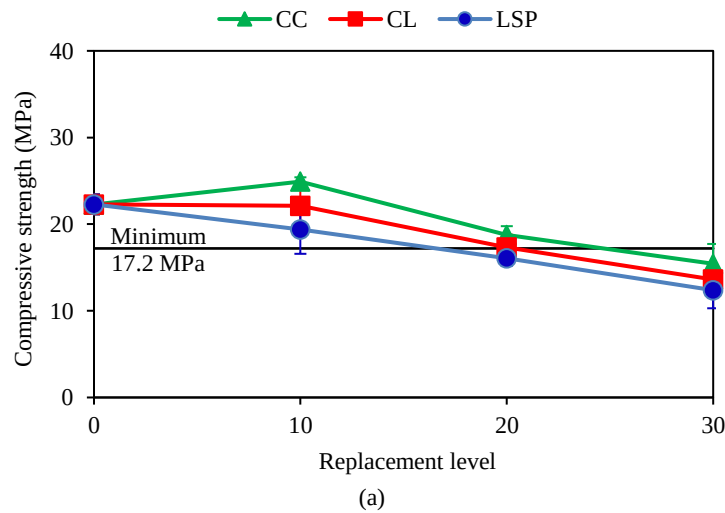


Figure 34: CO₂-actual, CO₂-th and carbonation efficiency of OPC and lime.

4.4.3 Characteristics of OPC-substituted mortars

Compressive strength

Figure 35 illustrates the compressive strength development of mortar samples containing CC, CL, and LSP at different substitution percentages over a curing period of 3, 7, and 28 days. Following the same paste samples behavior, the early age strength shows that both CC and CL begin to contribute positively to the strength development at 10% replacement level where CC mixture exhibited 24.91 MPa (111.8% of the control sample) followed by CL mixture which gave 22.11 MPa (99.24% of the control sample) while LSP came behind at 86.94%. This enhanced strength is due to the fine particles CL and CC accelerating the hydration reaction and providing additional sites for nucleation, leading to a higher early strength gain. However, as the curing period extends and the replacement level increases, a downward trend in strength is observed across all materials, with LSP showing the most significant reduction. By 28 days, while all mixtures retain compressive strengths above 17.2 MPa (minimum strength for standard mortar [295]), CC and CL mixtures show a notable stabilization in strength reduction (7.4% and 4.7%, respectively) compared to the control sample which exhibited 31.47 MPa. This strength reduction resulted from reduced cement content (dilution effect), translating to fewer hydration products that contribute to ultimate strength [296].



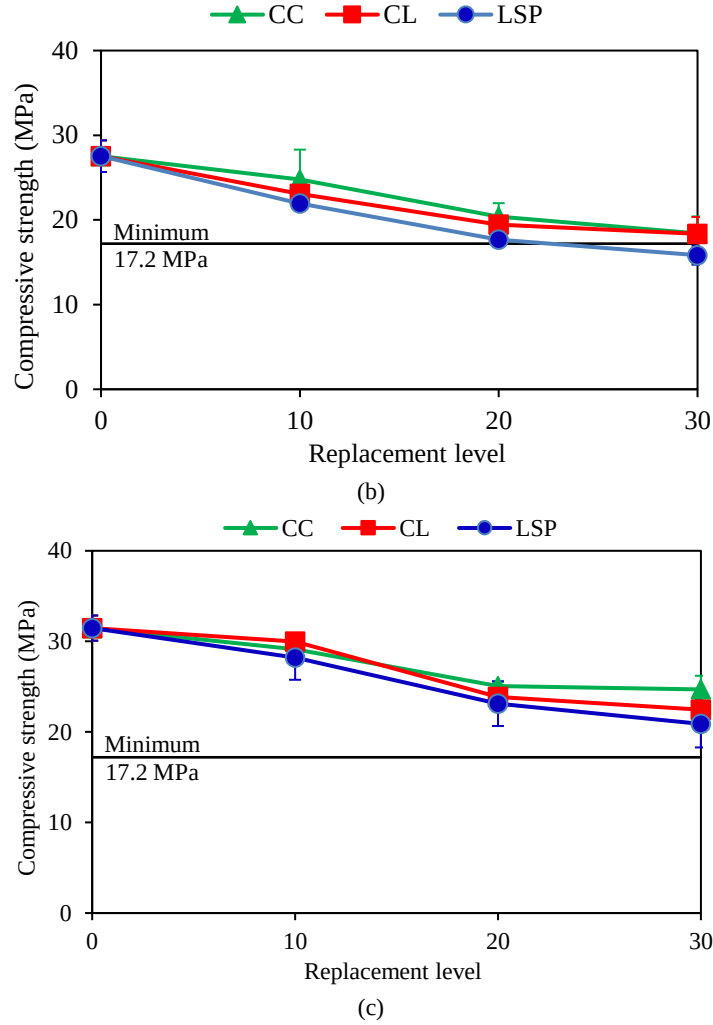


Figure 35: Compressive strengths of cement mortars incorporating carbonated CRMs at: (a) 3 days, (b) 7 days, and (c) 28 days.

Splitting tensile strength

Figure 36 shows the 28-day splitting tensile strength of mortar samples with varying replacement levels of CC, CL, and LSP. The results show that CC and CL mixtures maintain a relatively stable tensile strength across the replacement levels, maintaining 94.34% and 91.27% of the control sample's strength (which was 4.24 MPa) at 10% replacement, and gradually decreasing to 87.5% and 85.14% at 30% replacement, respectively. In contrast, the splitting tensile strength of LSP mixtures shows a more significant decrease with increasing replacement levels, starting from 86.56% of the control sample's strength at a 10% replacement level and dropping to 72.64% at the highest replacement level. This decline is attributed to its larger particle size compared to carbonated materials. This behavior aligns with the trends observed in compressive strength development and indicates that while LSP can be used as a partial replacement, its negative impact on tensile strength is considerably considered, especially at higher replacement percentages.

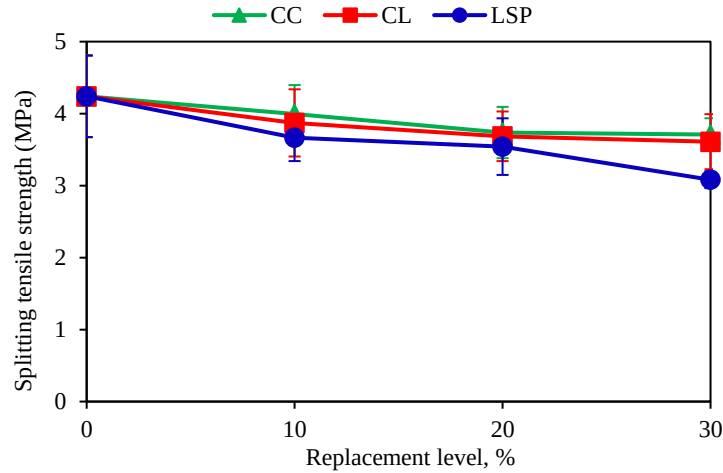


Figure 36: 28-Days splitting tensile strength of cement mortars incorporating carbonated CRMs.

Modulus of elasticity

Figure 37 presents the 28-day elastic modulus of mortar samples with varied proportions of CC, CL, and LSP. The data indicates that the control mixture demonstrated an elastic modulus of 33.58 GPa. At low replacement levels, the CC and CL mixtures achieved 99% and 95%, respectively, of the control mixture's elastic modulus, whereas the LSP variant attained only 84%. When the replacement level was increased from 20% to 30%, the elastic modulus of the CC and CL mixtures demonstrated a moderately reduced elastic modulus, retaining between 76-79% and 75-70% of the control mixture's modulus, respectively. In contrast, the LSP mixtures exhibit a more pronounced decline in their modulus values, decreasing from 75.2% at a 20% replacement level to 59.7% at a 30% replacement level.

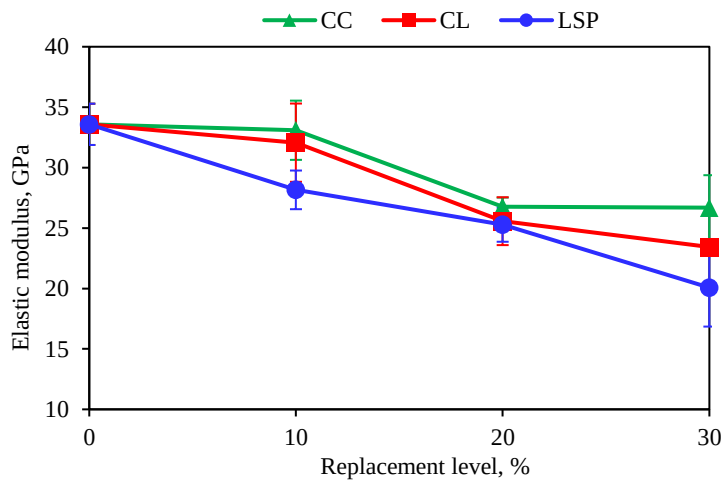
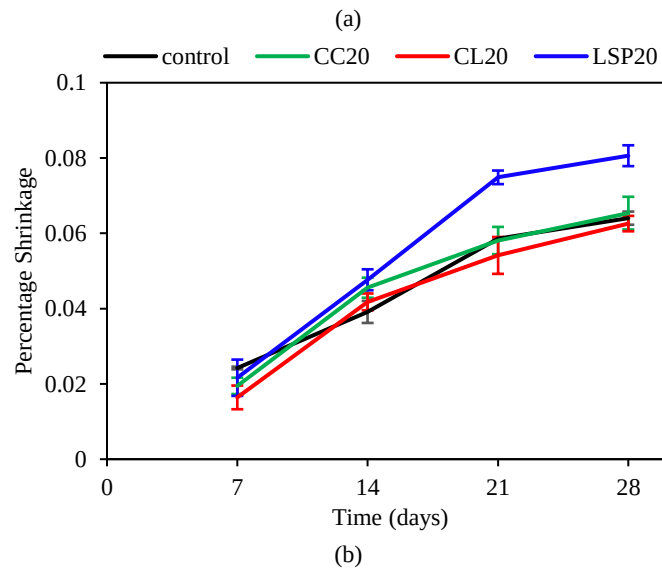
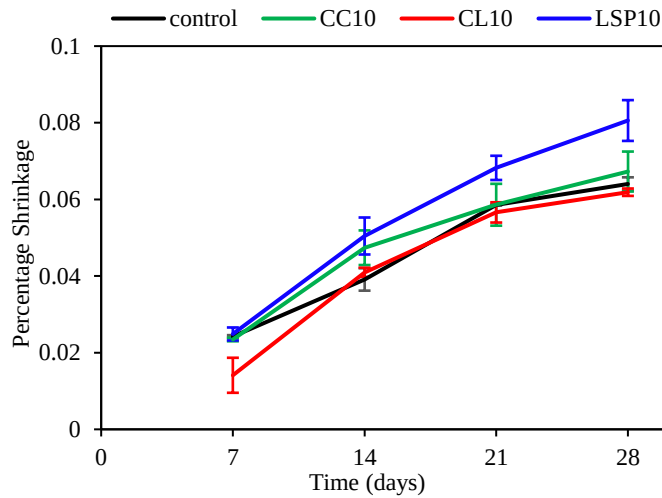


Figure 37: 28-Days elastic modulus of cement mortars incorporating carbonated CRMs.

Drying shrinkage

Figure 38 shows Drying shrinkage tests results. The incorporation of carbonated materials into mortar does not increase drying shrinkage, particularly at a 30% replacement level. This reduction is likely due to the high fineness of the synthesized carbonated materials providing a filling effect, reducing pore space, which can mitigate shrinkage. Conversely, mortar samples with LSP demonstrate notably higher drying shrinkage, which can be ascribed to its coarser particle size distribution, potentially increasing the void ratio and thus susceptibility to shrinkage during drying.



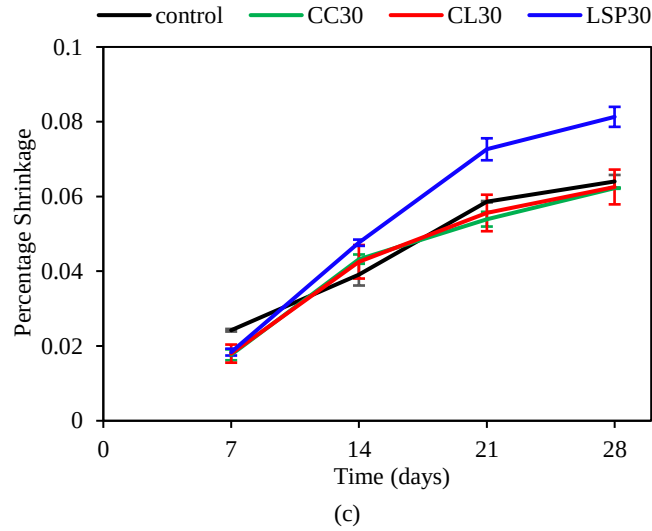


Figure 38: Drying shrinkage of cement mortars incorporating carbonated CRMs at: (a) 10, (b) 20, and (c) 30% replacement level.

Water absorption

Figure 39 shows 28-days water absorption against the level of replacement. The control mixture demonstrates a water absorption value of 7.55%. A general trend observed in the data is the incremental rise in water absorption corresponding to increased replacement levels. However, this trend was not universal, as evidenced by the CC mixture, which demonstrated a decrease in water absorption, from 8.5% to 8.24%, as the replacement level increased from 20% to 30%. Overall, all mixtures exhibited water absorption values ranging from 7.55 to 8.86% across all replacement levels, which are within the acceptable limits for mortar [297].

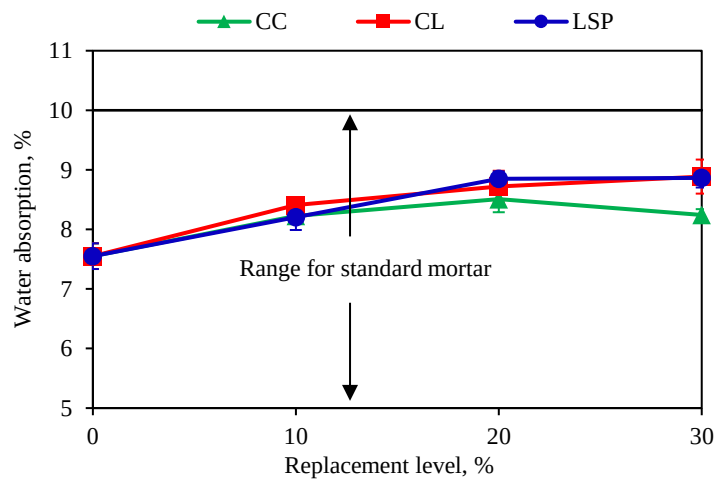


Figure 39: 28-Days water absorption of cement mortars incorporating carbonated CRMs.

Chloride migration

Figure 40 shows 28-day chloride migration coefficient versus the level of replacement. The control mixture demonstrates a chloride migration coefficient of $13.88 \times 10^{-12} \text{ m}^2/\text{s}$. Notably, at low replacement levels, all mixtures maintained coefficients remarkably close to the control value. However, when the replacement level increases the chloride migration increases. This is due to the reduced ability of carbonated CRMs to bind chloride ions. Normally, cement hydration products chemically bind chlorides, but this binding ability is reduced when cement is partially replaced [180,181]. Interestingly, CC mixtures exhibited the most minimal increase in chloride migration coefficient across all tested mixtures, even at the highest replacement levels, recording a value only 1.28 times that of the control mixture. At a 20% replacement level, CL mixture performed better than the LSP mixture, with coefficients of 1.19 and 1.33 times that of the control mixture, respectively. However, this trend reversed at the highest replacement level, where the LSP mixture's coefficient increased to 60% above the control mixture's, surpassing the CL mixture which showed an increase of 88%.

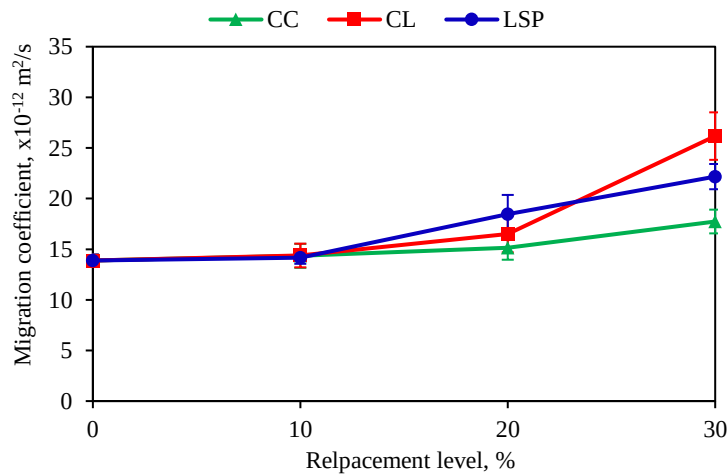


Figure 40: 28-Days chloride migration of cement mortars incorporating carbonated CRMs.

4.4.4 Characteristics of OPC-substituted concrete

Based on the findings from blended mortars characteristics, which indicated an adequate level of mortar's strength/durability can be achieved at the intermediate replacement level, a 20% replacement of OPC with carbonated CRMs was selected for concrete production in this study.

Compressive strength

Figure 41 presents the compressive strengths of concrete mixtures incorporating carbonated CRMs. The control mixture exhibited a compressive strength of 35.07 MPa.

Remarkably, concrete mixtures incorporating CC and CL demonstrated compressive strengths exceeding 30 MPa, corresponding to 96% and 94% of the control mixture's strength, respectively. In contrast, the mixture containing LSP did not reach 30 MPa, maintaining only 79% of the control mixture's compressive strength. These findings imply that the incorporation of chemically synthesized carbonated materials in concrete production can outperform the natural calcite source in preserving compressive strength property.

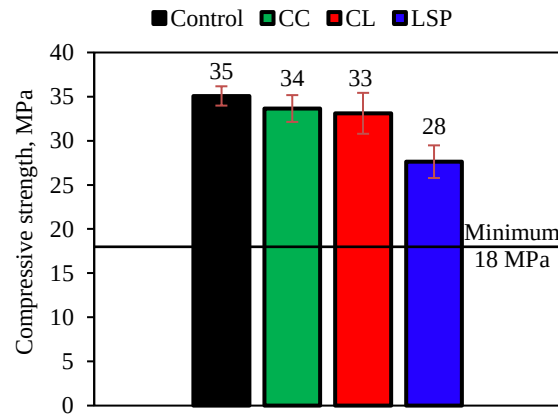


Figure 41: 28-Days compressive strengths of concrete samples incorporating carbonated CRMs at 20% replacement level.

Modulus of elasticity

Figure 42 presents the modulus of elasticity of concrete mixtures incorporating carbonated CRMs. The control mixture showed an elastic modulus of 34.1 GPa. In a similar pattern to compressive strength findings, concrete mixtures incorporating CC and CL performed better than LSP mixture and maintained 93% and 86% of the control mixture's value, respectively, in contrast to the LSP mixture, which retained only 72%. This reduction in elastic modulus is attributed to the low specific gravity of CRMs.

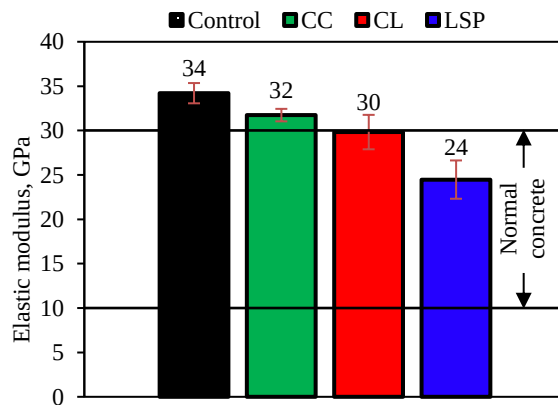


Figure 42: 28-Days elastic modulus of concrete samples incorporating carbonated CRMs at 20% replacement level.

Water absorption

Figure 43 shows the water absorption of concrete samples made with carbonated CRMs. The control mixture demonstrates a water absorption value of 3%. Notably, the concrete mixtures containing CC and CL demonstrated superior performance in comparison to LSP mixture. Specifically, CC and CL mixtures exceeded the control mixture's water absorption value by 14% and 12% respectively, while the mixture incorporating LSP exhibited the highest water absorption rate among the tested samples, reaching 125% of the control mixture's value. This indicates a superior water absorption resistance in mixtures containing CC and CL as compared to those with LSP.

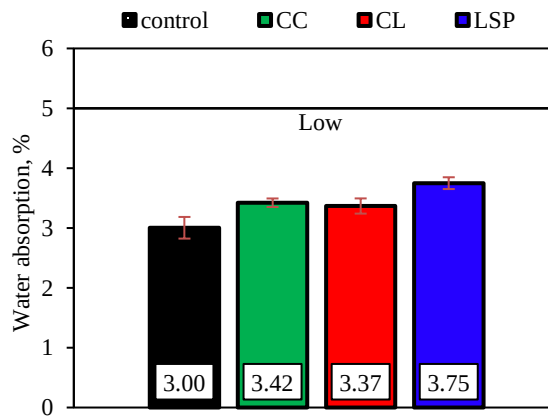


Figure 43: 28-Days water absorption of concrete samples incorporating carbonated CRMs at 20% replacement level.

4.4.5 Environmental impact assessment

Table 15 and Table 16 show the typical E-CO₂ and EE factors for CRMs used in this study, associated with modules A1, A2, and A3 (Figure 44). These modules are very likely to constitute the majority of E-CO₂ and EE over the life cycle of a structural project [285]. Additionally, the actual CO₂ uptake and energy consumption through the carbonation of CRMs were estimated in order to determine the net E-CO₂ and EE.

The total net amount of E-CO₂ and EE of mortar/concrete mixes can be estimated by multiplying the weight of each material (for every cubic meter) by its corresponding E-CO₂ and EE factor, and then summing the results (Table 17, Figure 45).

Generally, mixes containing carbonated CRMs exhibited slightly lower E-CO₂/m³ values than the control mixture, and mixtures containing LSP had significantly lower E-CO₂/m³ values. This is because the E-CO₂ factors of OPC and lime are higher than LSP. Therefore,

using CRMs with relatively lower E- CO₂ factors, such as Class C fly ash (0.008 kg/kg) [285], can be used in the application of this method to reduce the environmental impact.

On the other hand, mortar mixes containing carbonated CRMs exhibited higher E-CO₂/MPa values than the control mixture, and these values increased as the replacement level increased. This indicates that producing mortar that incorporates carbonated CRMs to achieve a specific strength, results in higher E-CO₂ values compared to plain mortar. This is primarily due to the reduced compressive strength of these blends and the relatively high E-CO₂ factors of the selected CRMs. However, the situation is different for concrete. The incorporation of CRMs in concrete mixes maintained compressive strength more effectively than in mortar mixes, resulting in a lower E-CO₂/MPa for the blended mixes compared to the control.

Furthermore, the process of carbonation was observed to have a minimal impact on the EE of mixtures made with carbonated materials, indicating that while carbonation effectively reduces E-CO₂, its influence on energy consumption is relatively minor.

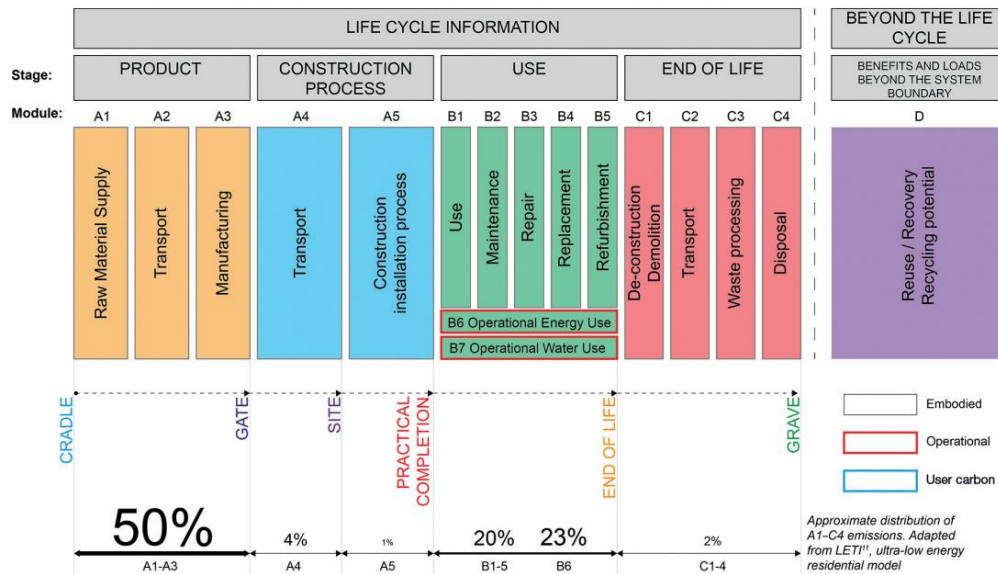


Figure 44: Lifecycle stages and modules [285].

Table 15: Inventory of E-CO₂ [285].

Raw material	OPC	Lime	LSP	FA	CA	Water
E-CO ₂ (kg/kg)	0.912	0.78	0.032	0.00493	0.004	0.0008
CO ₂ uptake (kg/kg)	0.22	0.41				
Net E-CO ₂ (kg/kg)	0.692	0.37	0.032	0.00493	0.004	0.0008

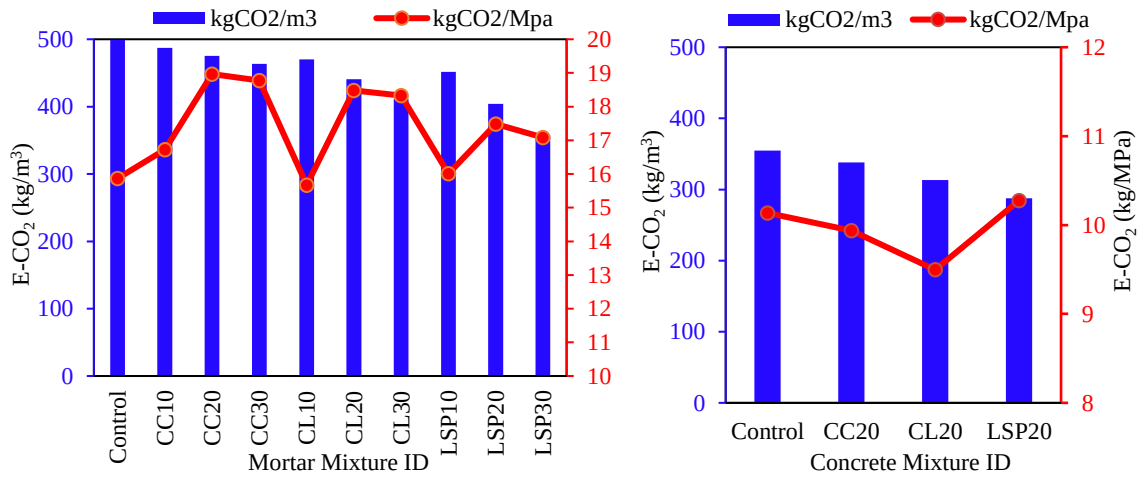
Table 16: Inventory of EE.

Raw material	OPC	Lime	LSP	FA	CA	Water
EE (MJ/kg)	4.53 [298]	4.25 [299]	0.09 [299]	0.02 [298]	0.1 [298]	0 [298]
Energy consumption (MJ/kg) ⁽¹⁾	0.04	0.05				
Net EE (MJ/kg)	4.57	4.30	0.09	0.02	0.1	0

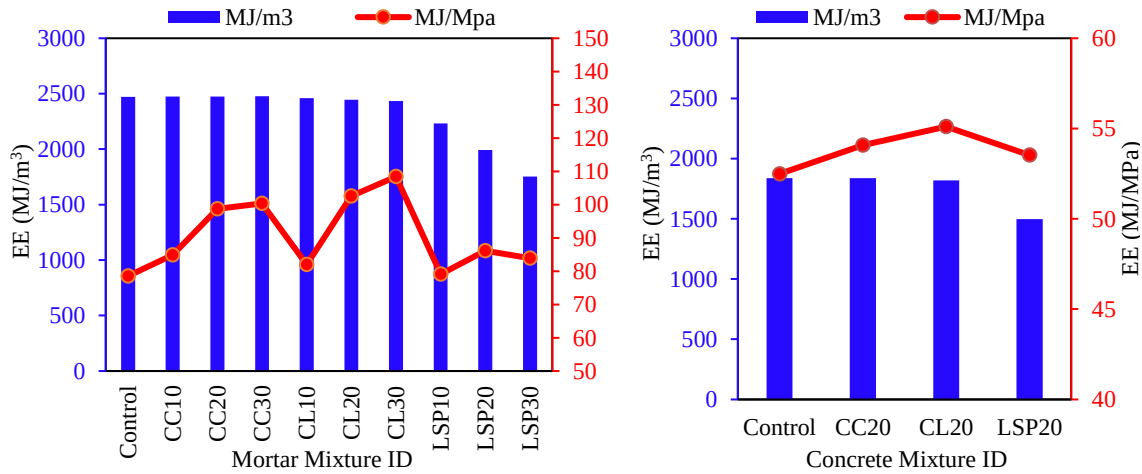
⁽¹⁾ Based on mixer's specifications

Table 17: E-CO₂ and EE of mortar/concrete mixtures.

Mixture ID	Net E-CO ₂				Net EE			
	kgCO ₂ /m ³		kgCO ₂ /MPa		MJ/m ³		MJ/MPa	
	Mortar	Concrete	Mortar	Concrete	Mortar	Concrete	Mortar	Concrete
Control	499.13	354.68	21.30	10.13	2471.44	1837.18	105.48	52.49
CC10	487.27	-	23.26	-	2473.45	-	118.09	-
CC20	475.41	337.88	23.33	9.94	2475.47	1838.86	121.46	54.08
CC30	463.55	-	27.72	-	2477.48	-	148.16	-
CL10	469.92	-	21.01	-	2459.03	-	109.95	-
CL20	440.70	313.41	22.77	9.50	2446.62	1818.53	126.40	55.11
CL30	411.48	-	22.42	-	2434.22	-	132.60	-
LSP10	451.70	-	25.00	-	2232.11	-	123.53	-
LSP20	404.26	287.72	23.07	10.28	1992.78	1498.58	113.74	53.52
LSP30	356.83	-	28.01	-	1753.45	-	137.63	-



(a) E-CO₂ analysis



(a) EE analysis

Figure 45: E-CO₂ and EE for mortar/concrete mixtures.

4.5 Comparative Analysis of Proposed Carbonation Approach and Existing Technologies

This section revisits various carbonation technologies previously introduced, which have been developed to enhance CO₂ sequestration in cast-in-place concrete. These include pre-carbonation, carbonation mixing (gas and liquid CO₂ forms), and the novel approach developed in this study. The methodologies, effectiveness, and practical applications of these three approaches are compared, with a summary provided in Table 10.

4.5.1 Carbonation procedure

Pre-carbonation involves a two-stage process where CO₂ is first injected into a slaked lime slurry with a 1% concentration, and then mixed with other concrete ingredients [18]. On the other hand, the carbonation mixing technique injects CO₂ directly during the mixing phase, either in gas form [202] or liquid form [203,204]. The approach suggested here involves injecting CO₂ into a CRM slurry, which is then processed into a powdered form before being blended into OPC-based systems.

4.5.2 CO₂ sequestration efficiency

Carbonation technologies are pivotal in reducing the carbon footprint and enhancing material performance within the concrete industry. A significant sustainability metric for these technologies is the CO₂ captured per megapascal (C-CO₂/MPa), which quantifies the CO₂ captured relative to the strength of the carbonated OPC-based mixture. Although these technologies are implemented at different phases of OPC-based systems and direct comparisons may be challenging, this analysis offers a broad comparison of potential CO₂ absorption capacities.

For pre-carbonation method, determining the C-CO₂/MPa starts by calculating the embodied CO₂ per megapascal (E-CO₂/MPa). E-CO₂/MPa was calculated following the same procedure in section 4.4.5. The C-CO₂/MPa, as illustrated in Figure 28, is then calculated by subtracting the E-CO₂/MPa of the CO₂-treated mortar mix from that of the control mix (prepared without carbonation treatment).

The evaluation did not further consider gas-based CO₂ mixing due to its failure to retain satisfactory strength. A life cycle assessment of CarbonCure technology indicated that it could sequester an average of 15 kg CO₂/m³ of concrete [286]. Given a compressive strength of 28.3 MPa achieved through this technology [205], the C-CO₂/MPa was estimated at around 0.53 kg CO₂/MPa, as shown in Figure 46.

Following the same analysis method used for pre-carbonation, the C-CO₂/MPa for concrete mixes incorporating 20% carbonated CRMs was calculated. As shown in Figure 46, the proposed methodology came second after pre-carbonation method in terms of C-CO₂/MPa. Notably, mixes containing carbonated CC showed the lowest C-CO₂/MPa value relative to those with CL. This is attributed to the high E-CO₂ factors of OPC. To mitigate this, utilizing CRMs with relatively lower E-CO₂ factors, such as Class C fly ash, could reduce the environmental impact when applying this method.

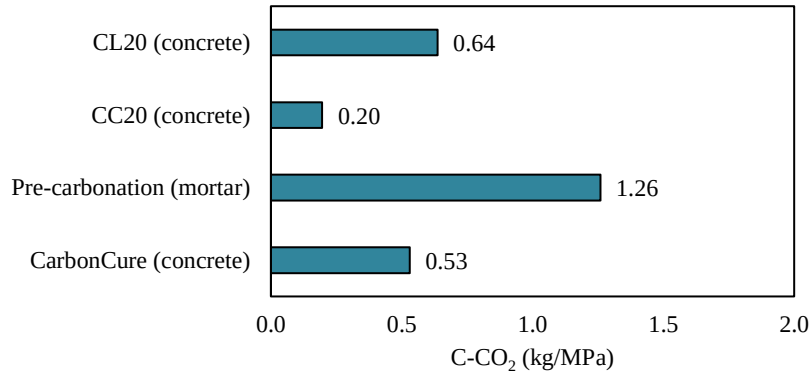


Figure 46: C-CO₂ analysis of recent carbonation technologies.

4.5.3 Impact on strength

The influence of carbonation technologies on concrete's mechanical properties varies significantly. Pre-carbonation method showed an increase in 28-day compressive strength by 16%, while the gas-based CO₂ mixing significantly reduced paste's strength by 56% [202]. CarbonCure technology (using liquid CO₂), however, claims to maintain the strength of concrete even with a binder content reduction of up to 7% [205].

The approach proposed in this study showed a 6% reduction in the compressive strength of concrete incorporating 20% carbonated CRMs. It is important to note that pre-carbonation method, with its 1% replacement, and gas CO₂ mixing, with significant strength reduction, present limitations in their applicability and sustainability impacts. Additionally, it is critical to emphasize that the proposed method not only may sequester more CO₂ compared to the CarbonCure approach, as mentioned in section 3.5.2, but also promotes the use of waste materials. This dual benefit underscores its potential for sustainability, aligning it more closely with environmental goals.

4.5.4 Practical applications and market viability

Unlike pre-carbonation and carbonation mixing, which require specific CO₂ injection setups and are challenging to adapt for on-site use due to logistical constraints, the method developed in this study improves practicality and scalability. It allows the production of dry powdered carbonated CRMs that can be easily transported, stored, and used in construction, similar to other building materials, showcasing significant market potential.

Table 18: Comparative Summary of recent carbonation technologies for cast-in-place concrete (adapted from [18,202,205,206])

Feature	Pre-carbonation	Gas CO ₂ mixing	CarbonCure	Proposed method
Carbonation methodology	Pre-carbonating lime slurry (1% concentration).	CO ₂ injection during mixing.	CO ₂ injection during batching.	CO ₂ injection into CRM slurry, followed by drying and powdering.
CO ₂ state	Gaseous.	Gaseous.	Liquid.	Gaseous.
Tested phase	Mortar.	Paste.	Concrete.	Concrete.
28-Days strength increase	+16%.	-56%.	-7% binder. 100% strength.	-6% at 20% replacement.
Adaptability for on-site use	Requires CO ₂ setup.	Requires CO ₂ setup.	Requires liquid CO ₂ setup.	Easy transport and use of dry powder.

4.6 Conclusions

This investigation evaluates existing carbonation methods used in concrete and addresses their limitations by proposing a novel carbonation technique tailored for on-site concrete mixing. Using hydrated lime and ordinary Portland cement (OPC) as calcium-rich materials (CRMs), the method involves CO₂ injection into CRM slurries, powdering the carbonated products, and then incorporating them into concrete mixtures. Limestone powder (LSP) is employed as a natural calcium carbonate benchmark to compare the performance of the products – carbonated lime (CL) and carbonated cement (CC). This paper explores the production of these carbonated CRMs and examines their impact on the mechanical and durability properties of mortar and concrete. The study yields the following key findings:

Compressive strength test results demonstrate that, at a low replacement level, mortar samples with CC and CL outperformed LSP sample and achieved an early strength equivalent to the control sample. After 28 days, mortar samples with 10% CC, CL, and LSP preserved on average 96%, 95%, and 87% of the compressive strength and elastic modulus, respectively, in comparison to the control sample. At maximum replacement level, CC, CL, and LSP mixtures retained 79%, 71%, and 63% of these properties, indicating the superior performance of carbonated materials over LSP. In terms of tensile strength, CC and CL also demonstrated superior performance compared to LSP,

maintaining over 90% and 85% of the strength at the lowest and highest replacement levels, respectively, while LSP's values were at 86% and 73%.

Additionally, the drying shrinkage of mortar was evaluated, and the findings showcased that CC and CL reduced the drying shrinkage, likely due to their finer particle size and pore-filling effects compared to LSP. As for water absorption, all mixtures showed an increasing trend as replacement level increases, ranging from 7.6% to 8.9%. Notably, the CC mixtures showed a decline in water absorption when replacement level increased from 20% to 30%. Regarding chloride migration, all mixtures maintained coefficients almost similar to the control mixture at low replacement level. Nevertheless, CC mixtures exhibited the most insignificant rise in chloride migration coefficient with a value only 1.3 times that of the control mixture at maximum replacement level.

A 20% replacement level was identified as the optimal balance for achieving maximal CO₂ sequestration without compromising the strength and durability of the mortar. Consequently, concrete mixtures were formulated with this replacement level. Findings showed that CC and CL had a favorable effect on compressive strength, elastic modulus and water absorption compared to LSP.

Furthermore, sustainability analysis was carried out on the produced mortar/concrete mixtures to evaluate the impact of the carbonation method on E-CO₂ and EE as key sustainability indicators. It was found that mixtures containing CRMs generally have lower E-CO₂ values compared to the control mixture, with mixtures containing LSP showing the most significant reduction. This reduction is attributed to the higher E-CO₂ factors of OPC, and lime compared to LSP. However, the E-CO₂/MPa was higher in blended mortar mixes than in the control. This is attributed to the reduced compressive strength of these mixes and the relatively high E-CO₂ factors of the CRMs used. In contrast, CRM incorporation in concrete (as opposed to mortar) effectively maintained compressive strength, thereby reducing E-CO₂/MPa of blended mixes below that of the control. Remarkably, carbonation had a negligible effect on the EE of the mixtures produced with carbonated materials.

A comparative study of established carbonation methods in concrete highlighted diverse outcomes. Pre-carbonation and gas-based CO₂ mixing faced challenges, the former due to minimal replacement rates and the latter due to compromised strength. Liquid-based CO₂ mixing preserved strength while achieving moderate CO₂ capture. The proposed strategy involving carbonated calcium-rich materials (CRMs) at minimal replacement levels stood out for its superior CO₂ capture and practicality, while also preserving satisfactory strength.

4.7 Recommendations

The suggested approach effectively captures CO₂, offering environmental benefits. In this study, pure CO₂ was used, however, for practical purposes, flue gas might serve as a more economical source of CO₂. OPC and lime were employed as foundational materials to test

the proposed carbon utilization technique in cement-based composites. Sustainable alternatives such as Class C fly ash and slag could replace OPC and lime in real applications of this carbonation process. Waste cement from washouts in ready-mix plants closely resembles the 1-hour hydrated OPC used in this study. Consequently, the results pertaining to the carbonated OPC provide crucial insights for the industrial recycling of waste cements into environmentally beneficial, carbon-sequestering construction materials.

4.8 Acknowledgements

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4.9 CRediT Author Contribution Statement

Omer Ahmed: Formal analysis, Investigation, Data Curation, Writing – Original Draft, Writing – Review & Editing, Visualization. **Shamsad Ahmad:** Conceptualization, Methodology, Validation, Writing – Review & Editing, Supervision. **Saheed Kolawole Adekunle:** Conceptualization, Methodology, Validation, Resources, Writing – Review & Editing, Visualization, Supervision, Project administration, Funding acquisition. **Mohammed Ali Alosta:** Validation. **Mesfer Mohammad Al-Zahrani:** Validation. **Ahmed Abd El Fattah:** Validation.

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

The study presented evaluates recent carbonation techniques in concrete and addresses their key limitations by introducing an innovative off-site carbonation approach designed especially for on-site concrete mixing applications. Selecting hydrated lime and ordinary Portland cement (OPC) as calcium-rich materials (CRMs), the proposed approach involves injecting CO₂ into slurries of these CRMs, powdering the carbonated products, and then incorporating them into concrete mixtures. Limestone powder (LSP) was used to serve as a mineral carbonate benchmark to study the behaviors of the carbonated products – carbonated lime (CL) and carbonated cement (CC). The production and analysis of carbonated CRMs, and the effects of incorporating carbonated CRMs on the hydration kinetics, compressive strength and microstructure of cement paste, as well as mechanical and durability properties of mortar and concrete are discussed. The following conclusions can be drawn from the outcomes of this study:

Microstructure analysis confirms calcite as the dominant carbonation product of the CRMs. This is evidenced by DTG peaks and increased calcite peaks in XRD spectra. Changes in surface morphology observed in SEM micrographs revealed that carbonation of CRMs results in the formation calcite particles in OPC. In contrast, carbonation of lime shows both calcite and the less stable vaterite, as indicated by the earlier thermal decomposition of CaCO₃ in DTG curves.

Isothermal calorimetry results showed that incorporation carbonated CRMs alters hydration kinetics and heat evolution in paste mixtures. CL enhances reactivity due to its fine particle size, followed by CC, while LSP exhibited the lowest reactivity owing to larger particle sizes.

Paste compressive strength results highlighted that at 15% cement replacement, CL and CC enhanced early-age compressive strength by up to 5% and maintained more than 95% of the control mixture after 28 days. However, strengths generally decreased at the high replacement levels. Nonetheless, CL and CC maintained above 20 MPa strength in contrast to LSP mixtures that fell below 20 MPa strength.

The stated impacts of carbonated CRMs on strength were supported by TGA and XRD analyses. At a 15% cement replacement level, CC and CL enhanced the hydration products as indicated by more pronounced DTG and XRD peaks, whereas declines in those peaks were observed at 30% replacement.

Mortar compressive strength results demonstrated that, at a low replacement level, mixtures with CC and CL outperformed LSP mixture and achieved an early strength equivalent to the control mixture. After 28 days, mortar mixtures with 10% CC, CL, and LSP preserved on average 96%, 95%, and 87% of the compressive strength and elastic modulus, respectively, in comparison to the control mixture. At maximum replacement level, CC, CL, and LSP mixtures retained 79%, 71%, and 63% of these properties, indicating the superior performance of carbonated materials over LSP. In terms of tensile strength, CC and CL mixtures also demonstrated superior performance compared to LSP mixtures, maintaining over 90% and 85% of the strength at the lowest and highest replacement levels, respectively, while LSP's values were at 86% and 73%.

Drying shrinkage of mortar mixtures was evaluated, and the findings showcased that CC and CL reduced the drying shrinkage, likely due to their finer particle size and pore-filling effects compared to LSP. As for water absorption, all mixtures showed an increasing trend as replacement level increases, ranging from 7.6% to 8.9%. Notably, the CC mixtures showed a decline in water absorption when replacement level increased from 20% to 30%. Regarding chloride migration, all mixtures maintained coefficients almost similar to the control mixture at low replacement level. Nevertheless, CC mixtures exhibited the most insignificant rise in chloride migration coefficient with a value only 1.3 times that of the control mixture at maximum replacement level.

A 20% replacement level was identified as the optimal balance for achieving maximal CO₂ sequestration without compromising the strength and durability of the mortar. Consequently, concrete mixtures were formulated with this replacement level. Findings showed that CC and CL had a favorable effect on compressive strength, elastic modulus and water absorption compared to LSP.

Furthermore, sustainability analysis was carried out on the produced mortar/concrete mixtures to evaluate the impact of the carbonation method on E-CO₂ and EE as key sustainability indicators. It was found that mixtures containing CRMs generally have lower E-CO₂ values compared to the control mixture, with mixtures containing LSP showing the most significant reduction. This reduction is attributed to the higher E-CO₂ factors of OPC, and lime compared to LSP. However, the E-CO₂/MPa was higher in blended mortar mixes than in the control. This is attributed to the reduced compressive strength of these mixes and the relatively high E-CO₂ factors of the CRMs used. In contrast, CRM incorporation in concrete (as opposed to mortar) effectively maintained compressive strength, thereby reducing E-CO₂/MPa of blended mixes below that of the control. Remarkably, carbonation had a negligible effect on the EE of the mixtures produced with carbonated materials.

A comparative study of established carbonation methods in concrete highlighted diverse outcomes. Pre-carbonation and gas-based CO₂ mixing faced challenges, the former due to minimal replacement rates and the latter due to compromised strength. Liquid-based CO₂

mixing preserved strength while achieving moderate CO₂ capture. The proposed strategy involving carbonated calcium-rich materials (CRMs) at minimal replacement levels stood out for its superior CO₂ capture and practicality, while also preserving satisfactory strength.

5.2 Recommendations

The proposed method effectively utilizes CO₂ for environmental benefits. While pure CO₂ was used in this study, practical applications could leverage flue gas as a more cost-effective CO₂ source. OPC and lime were used in this work as a base to try out the proposed carbon utilization method in cementitious composites. Calcium-rich wastes, such as Class C fly ash and slag, can be a better sustainable alternative to replace OPC and lime in real-world applications of this carbonation technique. Waste cement recovered from plant and truck washouts in ready-mix plants is particularly close to the 1-hour hydrated OPC utilized in this study. Therefore, the findings reported herein for the carbonated OPC offer valuable information for industrial upcycling of waste cements into a carbon-trapping construction material that benefits the environment.

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