EFFECT OF ADDITION OF CARBON DIOXIDE DURING MIXING ON THE PROPERTIES OF CONCRETE PRODUCED USING OPC AND PPC

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Submitted by:

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DECLARATION

I hereby declare that the work presented in this dissertation entitled **"EFFECT OF ADDITION OF CARBON DIOXIDE DURING MIXING ON THE PROPERTIES OF CONCRETE PRODUCED USING OPC AND PPC"** submitted for the partial fulfilment of the requirements for the degree **of Master of Engineering** in **Infrastructure Engineering** at **Civil Engineering Department, Thapar Institute of Engineering and Technology, Patiala** is an authentic record of my work that is carried out under the supervision of **Dr. Manpreet Singh** (Assistant Professor, Department of Civil Engineering Thapar Institute of Engineering and Technology, Patiala Punjab). This work has not been submitted elsewhere to reward any other degree. I am fully responsible for the contents of my dissertation.

Dated: 29-07-2022

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CERTIFICATE

This is to certify that the above declaration made by the student concerned is correct to the best of my knowledge and belief.

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"It is not possible to prepare a project report without the assistance & encouragement of other people. This one is certainly no exception."

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ABSTRACT

Due to the modernization of the world, we live in, cement manufacturing has increased dramatically in recent years. Cement production is the third-largest source of anthropogenic carbon dioxide CO_2 emissions, behind fossil fuels and land-use change. Cement is the primary binding element in concrete, and as the concrete industry expands, so will CO_2 emissions. Carbon dioxide, or CO_2, is responsible for the majority of global greenhouse gas emissions. CO_2's capacity to last longer than other greenhouse gases and the ease with which it can be produced has resulted in a high yearly estimate. Sequestered CO_2 in concrete has the potential to reduce carbon emissions while simultaneously improving concrete's compressive strength. Carbon dioxide is sequestered during this procedure. As a result, reducing CO_2 output while keeping up with the concrete industry's advancement is critical in the current era. CO_2 sequestration has a role in this. It is a method in which CO_2 is transformed into a mineral that is permanently trapped in concrete. With the main ingredient responsible for global 7% carbon dioxide emissions and with no such material around having the same strength and durability properties, the concrete is the second-highest industrial source of carbon dioxide on the planet. While the Nations are agreeing to limit the rise in average global temperatures to 2ºC by taking immediate action to escalate cuts in carbon emissions at U N talks in 2010. The only way possible in achieving this goal is to sequester and reduce carbon emissions. And what else would be the best way to sequester and utilize the carbon dioxide emissions than to trap it in concrete forever. The carbon dioxide reacts with the hydrating cement to form calcium carbonate ($CaCO_3$), which is trapped inside the concrete and enhances the compressive strength and durability. This study aims to find the effect of carbon dioxide injection during mixing on the properties of the concrete. It is evident from the study that by utilization of the optimum dosage of carbon dioxide in the concrete, the concrete tends to densify and thus the best performance is achieved. Clearly the properties like fresh, mechanical, and durability all have the positive effect due to the CO_2 sequestration. Hence, it can be concluded that by the carbon dioxide sequestration we can save our planet by reducing the carbon footprint and also achieve the best performance economically.

Keywords- Sequestration, Concrete, Carbon dioxide, Durability, Microstructure.

Contents

LIST OF FIGURES

LIST OF TABLES

ABBREVIATIONS

CO_2	Carbon dioxide
GHG	Greenhouse gases
C_3S	Tri-calcium Silicate
C_2S	Di-calcium Silicate
CH	Calcium Hydroxide
$CaCO_3$	Calcium Carbonate
w/c	Water cement ratio
OPC	Ordinary Portland Cement
PPC	Portland Pozzolana Cement
RCPT	Rapid Chloride Permeability Test
UPV	Ultrasound Pulse Velocity
CC	Carbonation Curing
IS	Indian Standard
ASTM	American Society of Testing and Materials
CS	Compressive Strength
SEM	Scanning Electron Microscopy
XRD	X-Ray powder Diffraction

CHAPTER 1

Introduction

1.1 Greenhouse gas effect

The earth's atmosphere functions as a blanket to trap solar energy, heating and maintaining the earth's surface temperature. The average temperature of the earth's surface is roughly 15°C, but without the atmosphere, it might reach a surface temperature of -19°C, similar to that of the moon. There is no atmosphere to keep the moon's surface warm. The average surface temperature of the moon is roughly -23°C, despite the fact that it gets the same amount of solar energy as the earth. The greenhouse gas effect refers to the atmospheric phenomena that raises global temperatures by +34°C. The impact of greenhouse gases keeps the environment on earth habitable and safe for life (Kweku et al. 2018).

The atmosphere's rising carbon dioxide content is now disrupting the CO_2 greenhouse gases' natural composition as a result of human and anthropogenic activity. Furthermore, some contend that the increase in atmospheric CO_2 is driving a rise in world temperatures. As the temperature rises, more water vapour, a greenhouse gas, is released into the atmosphere. Most scientists believe that the globe is warming faster than it has in the previous 10,000 years, and that this warming is being driven by rising levels of carbon dioxide and other greenhouse gases in the atmosphere. An increase in global temperature is likely to have a wide range of possible impacts and repercussions. Due to melting glaciers, melting Antarctic ice caps, and thermal expansion of ocean water, the ocean water level is predicted to rise and threaten many coastal communities with flooding. Desertification is projected to be a major trend in the tropical zone. Global warming has a significant impact on people and wildlife, disrupting a viable and comfortable environment(Clark and York 2005).

Carbon dioxide (CO_2) is the most significant anthropogenic greenhouse gas (GHG), accounting for 63.9% of the increased greenhouse effect. Other greenhouse gases produced by anthropogenic activity include methane (CH_4), nitrous oxide (N_2O), ozone (O_3), chlorofluorocarbons (CFCs), and fluorocarbons (CFs). Figure 1.1 depicts the relative contribution of various gases to climate change.

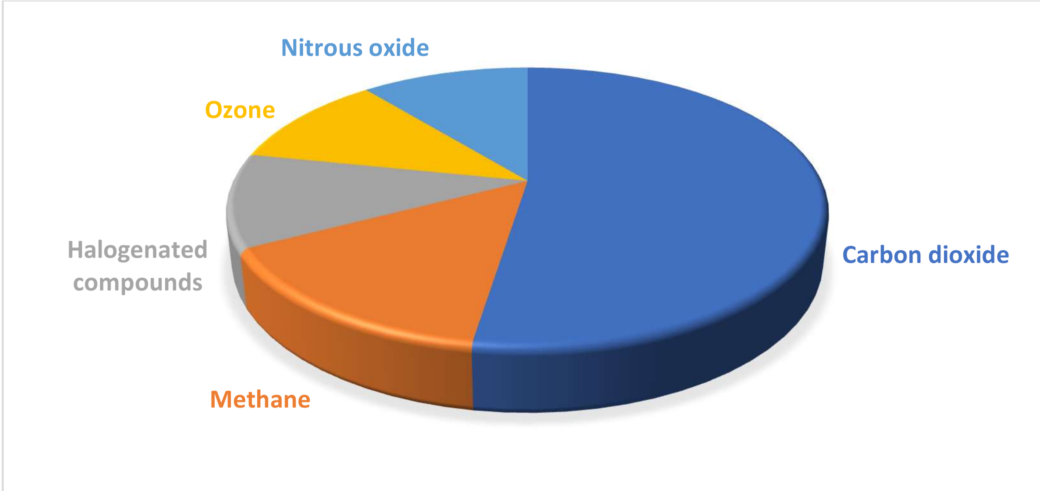


Figure 1. 1 Contribution of Greenhouse gases to climate change (Source: World Meteorological Organization)

1.2 Sources of carbon dioxide

To avoid catastrophic climate change, we must drastically cut global greenhouse gas emissions. Every year, the globe emits around 50 billion tonnes of greenhouse gases [measured in carbon dioxide equivalents (CO_2eq)].

To determine how to cut emissions most efficiently and which emissions can and cannot be removed using present technology, we must first identify where our emissions originate. Figure 1.2 depicts the global breakdown of greenhouse gas emissions. This most recent sector-by-sector summary of global emissions was released by the World Resources Institute and Climate Watch (Watch 2020). It can be said that over three-quarters of emissions are caused by energy usage, nearly one-fifth by agricultural and land use, and the remaining 8% by industry and waste.

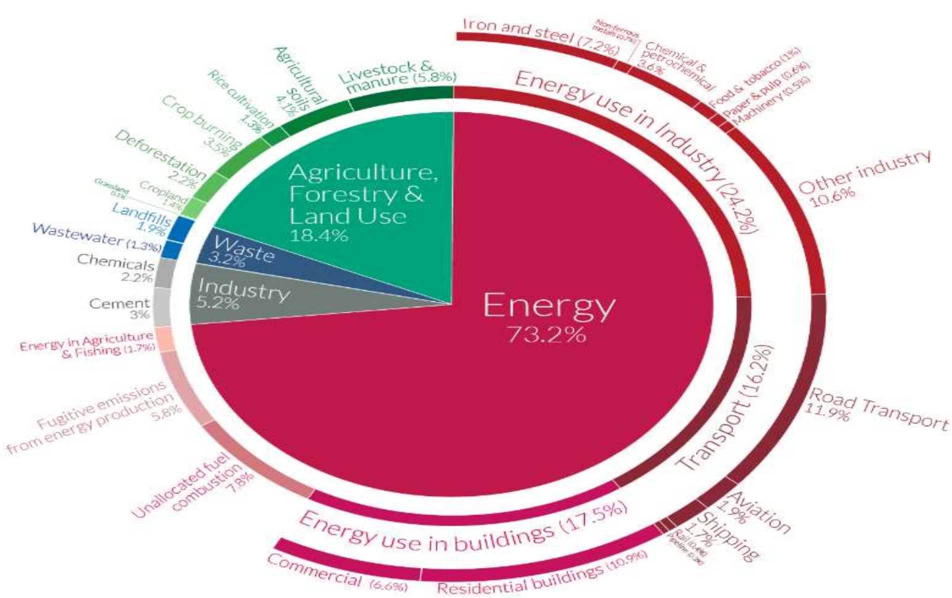
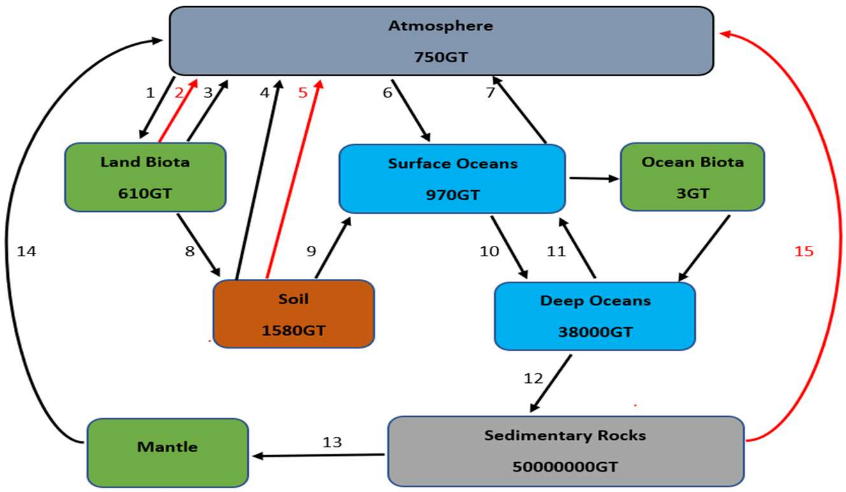


Figure 1. 2 Global Greenhouse gas emissions by sector (Source: Climate Watch and World Resources Institute)



→	Related to Human Activities	1	Photosynthesis
2	Burning	3,4	Respiration
5	Farming	6,7	Ocean-Atmosphere Diffusion
8	Litter Fall	9	Runoff
10,11	Upwelling & Downwelling	12	Sedimentation
13	Subduction	14	Volcanic Eruptions
15	Fossil Fuel Burning	GT	Gigatons = 10^{15}g

Figure 1. 3 Global Carbon Cycle (Source: The Blue Planet Company)

1.4 Role of concrete in greenhouse gas emissions

Global warming is a major issue for all countries in the current scenario. Despite the absurd discussions over the presence of global warming, scientific study has consistently demonstrated the effects of global warming and how they will worsen in the future. The fundamental cause of global warming is undeniably greenhouse gases. Out of all the greenhouse gases created, carbon dioxide significantly impacts the world we live in. CO_2's capacity to persist longer than other greenhouse gases and the ease with which it is produced has led to its high annual forecast. Humanity has sequestered 30 million tonnes of CO_2 during the previous 40 years, whereas the entire atmosphere has 750 gigatons of CO_2; hence, CO_2 sequestration has been nil in comparison.

One of the significant contributors to climate change is right beneath our feet, and transforming it could be a powerful solution to keep greenhouse gases out of the atmosphere. The concrete has an emission problem. The energy-intensive process of making concrete releases a massive amount of CO_2 into the atmosphere. Its main ingredient (cement) is responsible for approximately 7% of global CO_2 emissions (Andrew 2018). Concrete is the second most consumed substance on the planet after water (Santhanam, Ramesh, and Pohsnem 2020). Concrete is a synthetic rock made with a mixture of stones, sand, water, and most importantly, cement to bind it all together. But cement has a massive carbon footprint. One pound of cement releases one pound of CO_2 emissions. It is the second-highest industrial source of CO_2 on the planet (Emetere and Dania 2019). There are two stages to the cement manufacturing process that result in CO_2 emissions. The first phase is the compound response, which occurs when the major concrete elements, such as clinker and carbonates (most commonly limestone, $CaCO_3$), decompose into oxides (CaO). According to studies, this process contributes roughly 5% of anthropogenic CO_2 fluxes. The CO_2 combustion process is the second source of emissions. This occurs when fossil fuels are burned to create the substantial energy required to heat the basic materials to temperatures considerably above 1000°C (Andrew 2018). It is a threat to the earth but also an opportunity, indicating that cement is a large, fat target for technical advancement.

As a result, progress has been made towards developing the next generation of concrete technology capable of lowering the carbon footprint of infrastructure and increasing its longevity. With the regular use of industrial by-products such as iron slag and coal fly ash to minimise the amount of cement required, the resulting concrete would emit much less carbon dioxide. Additionally, other binders such as calcined limestone clay can be utilised to minimise the cement concentration. According to one study, using limestone and calcinated clay can help reduce emissions by at least 20% while also lowering production costs. Apart from producing blended cement, researchers and corporations are examining ways to incorporate collected CO_2 as an element in the concrete, encapsulating it and preventing it from escaping into the atmosphere. CO_2 can be added as particles – or as an injection during mixing. After the concrete has been cast, carbonation curing, commonly known as CO_2 curing, can be employed. These reactions convert CO_2 to a mineral, forming solid carbonates that may also strengthen concrete. This means that structures may use less cement, hence reducing associated emissions.

As a result, not only can CO_2 injection in concrete have a good environmental impact, but it may also be financially beneficial due to the prospect of injected concrete having superior strength and quality. Not only does the trapped CO_2 improve the properties of the concrete, but it also helps to reduce the amount of cement used in its production. These two selling aspects make this project appealing to the environment and positively impact the industry.

CO_2 sequestration in concrete can be achieved either by injecting CO_2 during the mixing stage or during the curing phase of the concrete. It is important to note that both have the same effect on concrete but at different levels or stages. Apart from these two methods, the CO_2 can be sequestered in the concrete through the carbonation of recycled aggregates.

1.5 Methods for utilization of Carbon dioxide in concrete

- **During the mixing stage:** In this stage, the CO_2 is injected into the concrete during batching. The metered supply of controlled CO_2 liquid is pressurized into the mixing concrete at a specified flow rate over a fixed injection interval. The liquid CO_2 is converted into the CO_2 gas and finely CO_2 particles called CO_2 snow. Hydrating cement releases calcium ions, which react with CO_2 to generate calcium carbonate.

- **During the curing stage:** Typical CO_2 curing of concrete is done in a sealed container. Afterward, a regulator controls the amount of CO_2 being pumped into the system. After 24 hours, the concrete is then placed into a curing tank.

- **Using Carbonated Aggregates:** The natural aggregates or the recycled aggregates are subjected to CO_2 curing before their utilization to enhance their properties and sequester the CO_2. The CO_2 curing of these aggregates is done in the sealed container with the controlled supply of CO_2 into the container and after the 24 hours, these aggregates can be utilized for their purpose.

From the above methods, the most amount of CO_2 is sequestered by the curing method and the best impact of CO_2 on concrete properties is when it is utilized during mixing stage.

1.6 Chemistry involved between concrete and carbon dioxide

Carbonation is a chemical reaction between cement binder and carbon dioxide in presence of moisture. During the mixing phase, after the addition of water and carbon dioxide, carbonation reaction occurs between cement anhydrate phases (C_3S or C_2S) and carbon dioxide in a fresh concrete immediately after formation. Its reaction is described by Equation. 1 and 2. Another type of carbonation reaction that takes place during curing phase is between hydration products, such as calcium hydroxide ($Ca(OH)_2$) and calcium silicate hydrate ($3CaO{\cdot}2SiO_2{\cdot}3H_2O$, or C-S-H), and carbon dioxide in a partially cured concrete within 24 hours. Equations 3 and 4 then govern the reaction. In both cases, the reaction products are primarily calcium carbonates and calcium silicate hydrates (Berger, Young, and Leung 1972).

$$2(3CaO{\cdot}SiO_2) + 3CO_2 + 3H_2O \rightarrow 3CaO{\cdot}2SiO_2{\cdot}3H_2O + 3CaCO_3 \qquad (1)$$

$$2(2CaO{\cdot}SiO_2) + CO_2 + 3H_2O \rightarrow 3CaO{\cdot}2SiO_2{\cdot}3H_2O + CaCO_3 \qquad (2)$$

$$Ca\,(OH)_2 + CO_2 \rightarrow CaCO_3 + H_2O \qquad (3)$$

$$3CaO{\cdot}2SiO_2{\cdot}3H_2O + 3CO_2 \rightarrow 3CaCO_3 + 2SiO_2 + 3H_2O \qquad (4)$$

As indicated by Equations (1)-(4), carbonation of concrete is a CO_2 sequestration process. Gaseous carbon dioxide is converted to thermodynamically stable calcium carbonate, which is embedded in calcium silicate hydrate. It was estimated that at full carbonation, all CaO reacts with CO_2 to form $CaCO_3$ (Steinour 1959).

The most advantageous method for utilising carbon dioxide in ready mixed concrete production involves adding carbon dioxide to fresh concrete during the batching and mixing process. A precise dose of liquid carbon dioxide is measured and administered in accordance with the batch's cement content.

This carbon dioxide experiences a phase shift following its passage through the flowmeter. This phase transition is visible as carbon dioxide is now a mixture of gaseous and solid snowy forms, which occurs just prior to the delivery of the concrete mixer. Carbon dioxide is added immediately after the water is mixed and is completed before the mixing is complete. When CO_2 gas is injected into fresh concrete from a gas cylinder, it changes its state to aqueous. The hydration of the gas will occur as a result of the reaction with water, resulting in H_2CO_3. The ionisation of H_2CO_3 produces H^+, HCO_3^-, and CO_3^{2-}. A coating of calcium-silicate-hydrate gel is then applied to the calcium silicate, which dissolves to release Ca^{2+} and SiO_4^- ions. CO_2 interacts with calcium ions in the solution to generate calcium carbonate ($CaCO_3$ nanoparticles). These nanoparticles have been shown to alter the initial stages of hydration, an adequate dose does not prevent future hydration products such as calcium hydroxide, ettringite, or calcium silicate hydrate gel from forming. Also, this in-situ nanoparticulate structure creation may aid in improving concrete's compressive strength.

Carbon dioxide reactivity with a mature concrete microstructure has been linked to concerns including shrinkage and pore solution reduction. Corrosion was triggered by pH and carbonation. While the carbonation process used in concrete manufacture uses CO_2 to react with hydrating cement in fresh concrete rather than the hydration phases found in mature concrete, the results are not the same.

Concrete carbonation is the reaction that occurs. Studies have indicated that carbonation harms the long-term growth of concrete, although the response that arises here is early carbonation. Carbonation from weathering has a deleterious impact on concrete, however, carbonation from a young age does not. As a result, the impact is different. When a fresh concrete mix is exposed to CO_2, early age carbonation occurs along with cement hydration. It improves the durability of cement-based materials, such as resistance to sulphate attack, chloride penetration, and freeze-thaw damage, by speeding up the early age strength growth of the concrete.

1.7 India's cement emission woes

Cement industry emissions in India have risen dramatically in recent decades, reaching a peak of 144 million metric tonnes of CO_2 in 2019. ($MtCO_2$). COVID-19, on the other hand, reduced emissions by 14 percent in 2020 to 123 $MtCO_2$. Despite these significant reductions, India remained the world's second-largest polluter from cement manufacture.

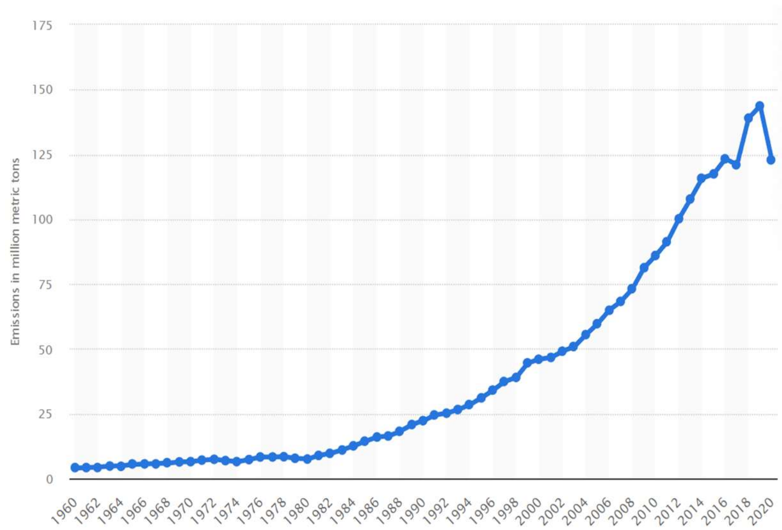


Figure 1. 4 CO_2 emissions from cement manufacturing in India 1960-2020 (Source: Statista)

The rise of India's industrial sector, with cement being the most manufactured product, is critical to the country's progress toward becoming a strong economy. In 2018-2019, India consumed 337 million tonnes of cement annually, which is predicted to rise to 550 million tonnes by 2025. This increased cement consumption is also associated with harmful greenhouse gas (GHG) emissions. The cement sector is solely responsible for 8% of total national emissions. The Indian cement industry's CO_2 emission intensity in 2018 was 576 kg CO_2/ton of cement produced, with the global average at 634 kgCO_2/ton of cement produced.

On the one side, cement is the most commonly used industrial material essential for development, but it also contributes significantly to GHG emissions. As a result, there is a need to strike a balance between national progress and environmental sustainability. Furthermore, in order to meet climate change mitigation targets in accordance with the Paris Agreement (UNFCCC 2016), which aims to limit global temperature increases by 2°C by the end of this century, there is an urgent need to investigate other options to limit GHG emissions from India's cement industry. Much research has recently gone into developing low-carbon cement alternatives, including LC^3, geopolymer binders, belite rich cements, and other innovative cement. Furthermore, novel technologies such as carbon capture and storage (CCS), renewable energy technologies, and others have the potential to cut emissions by 48% and must be widely adopted in the Indian cement sector. Even though these alternatives and technology increase the cost of cement manufacturing, it is a modest price to pay for the sake of the environment.

1.8 Research Objective

This research is primarily focused on sustainability and environmental benefits. The study's main objective is the sequestration and utilization of the carbon dioxide in the concrete. The carbon dioxide is injected into the concrete during the mixing phase. This research investigates the effect of the addition of various carbon dioxide dosages on the various properties of the concrete with different types of binders like OPC and PPC. Using carbon dioxide tends to increase the compressive strength of the concrete. Therefore, at the optimal dosage of CO_2, the cement quantity can be reduced as the desired strength can be achieved at the lower amount of cement. Thus, the optimal dosage of carbon dioxide required will also be investigated.

The main objectives of the study are as follows:

1. Study the effect of carbon dioxide addition in concrete on Mechanical properties - compressive strength and split tensile strength; Durability – RCPT, Water absorption, and Sorptivity.
2. To compare the mechanical and durability performance of OPC and PPC-based mixes.
3. To study the effect of carbon dioxide addition on the microstructure of concrete.

1.9 Organisation of the thesis

This thesis consists of five chapters and is organized in the following manner. **Chapter 1** is the Introduction, which highlights the role of carbon dioxide emissions in the degradation of the environment, especially those from the concrete industry. It presents the study on carbon dioxide sequestration in concrete and its mechanism. It also gives a brief about India’s cement emission, followed by the objectives of the thesis work. The remainder of the thesis is as follows. **Chapter 2** reviews the prior research on using carbon dioxide in concrete. All the methods of carbon dioxide utilization in concrete are reviewed with the main focus on using carbon dioxide during the mixing phase. Based on that, the necessity of present research has been identified. **Chapter 3** discusses the experimental samples' materials, design compositions, and the methodology adopted to conduct the experiments. **Chapter 4** discusses the results drawn from the experimental investigations on tests conducted on concrete. **Chapter 5** discusses the various conclusions drawn from this study's experimental investigations.

In the end, references used in this dissertation are presented.

CHAPTER 2

Literature Review

2.1 General

This chapter describes a state of art literature review on using carbon dioxide in concrete. The literature was reviewed for using carbon dioxide during the mixing phase, curing, and recycled concrete aggregates. However, the main focus was studying the effect of adding carbon dioxide during the mixing phase. This chapter also reviews the literature regarding carbonated concrete's fresh, mechanical, durability, and microstructural properties.

2.2 Effect of adding carbon dioxide

Various researchers have studied the use of carbon dioxide in concrete. The effect of adding carbon dioxide on the properties of concrete, as reviewed by multiple researchers, is discussed in the following section.

2.2.1 Fresh Properties

Studying the fresh properties of concrete is critical because they impact the quality of hardened concrete. The fresh properties include workability, setting time, and density. Amongst them, workability is an essential property of concrete that helps determine the ease with which it flows. As reported by the authors, the effect of the addition of carbon dioxide on the fresh properties of concrete is summarized below.

(Kamal et al. 2020)found that the concrete mixture exposed to carbon dioxide required more water than the one without exposure to carbon dioxide because of the reduction of water during the chemical reaction.

(Sean Monkman, Grandfield, and Langelier 2018)observed that injecting CO_2 into ready mixed concrete during its mixing and batching contributed to a slight increase in hydration and a prominent compressive strength increase due to the reaction of CO_2 with C_3S at the earliest stage of hydration, creating a nanoscale carbonate reaction product which accelerated the hydration. The time of set was reduced from 315 to 272 minutes.

(Samniang et al. 2021)prepared the mixture with and without fly ash replacement and then added pure CO_2 or exhaust gases (released from vans). It was concluded that the slump for mortar and concrete (without fly ash) is lower than the control. The slump of carbonated concrete declined, and the workability is reduced faster than the controlled one. While for PFA concrete (with fly ash), the slump of carbonated concrete was reduced with increasing the dosage of CO_2.

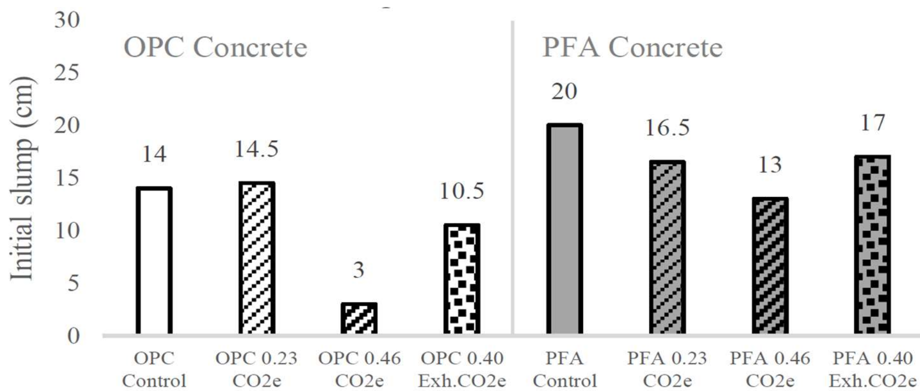


Figure 2. 1 Initial slump of concrete (Samniang et al. 2021)

(Sean Monkman, MacDonald, and Hooton 2016)compared the three mixes of concrete out of which one had CO_2 injected in it and found that injection of CO_2 into the concrete mixture accelerated the hydration and strength. Isothermal calorimetry revealed that the CO_2 injection accelerated early hydration reaction. Also, the mixture containing CO_2 showed significant improvement in it is compressive strength.

(Sean Monkman et al. 2016)prepared five concrete mixtures in which three mixtures were treated with increasing dose of CO_2. It was found that the CO_2 did not produce any change to the fresh concrete properties. Also, the CO_2 doses reduced the time of the initial set between 95 and 118 minutes (that is 22 to 25% reduction) and final set by 104 to 126 minutes (that is 21 to 25% reduction). The CO_2 dose of 0.15% provided the greatest acceleration benefit amongst the CO_2 -treated batches.

Table 2. 1 Time of set (Sean Monkman et al. 2016)

Batch	Initial set			Final set		
	Time (h)	Difference (min)	Relative to reference	Time (h)	Difference (min)	Relative to reference
Reference	7:08	-	100%	8:18	-	100%
Accelerated	4:15	-173	60%	5:36	-162	67%
CO_2-1	5:33	-95	78%	6:34	-104	79%
CO_2-2	5:10	-118	72%	6:12	-126	75%
CO_2-3	5:28	-100	77%	6:27	-111	78%

(Y. Wang, He, and Yang 2018)examined the effect of dry ice on the hydration and hardening behaviour of Portland cement and found that at dosages of dry ice less than 0.9 wt%, there was a very little effect on standard consistency and setting time but at dosages greater than 0.9% it increased rapidly. To be specific, it was found that at the dosage of 0.6 wt% the setting time is lowest with the initial setting time decreased by 12.3% and the final setting time reduced by 17.4%.

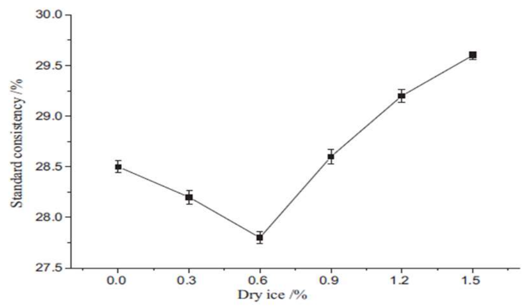


Figure 2. 2 Effect of dry ice on the standard consistency (Y. Wang, He, and Yang 2018)

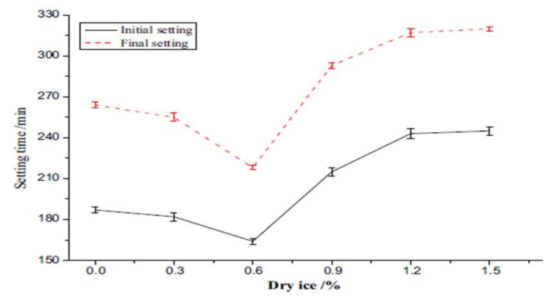


Figure 2. 3 Effect of dry ice on the setting time (Y. Wang, He, and Yang 2018)

(Samniang et al. 2021)prepared the mixture with and without fly ash replacement and then added pure CO_2 or exhaust gases (released from vans) in it. It was found that for mortar and concrete (without fly ash), the initial and final setting time of all specimens with CO_2 were longer than the ones without CO_2. While for PFA concrete (with fly ash), the initial and final setting time of the carbonated concrete is longer than that of the control.

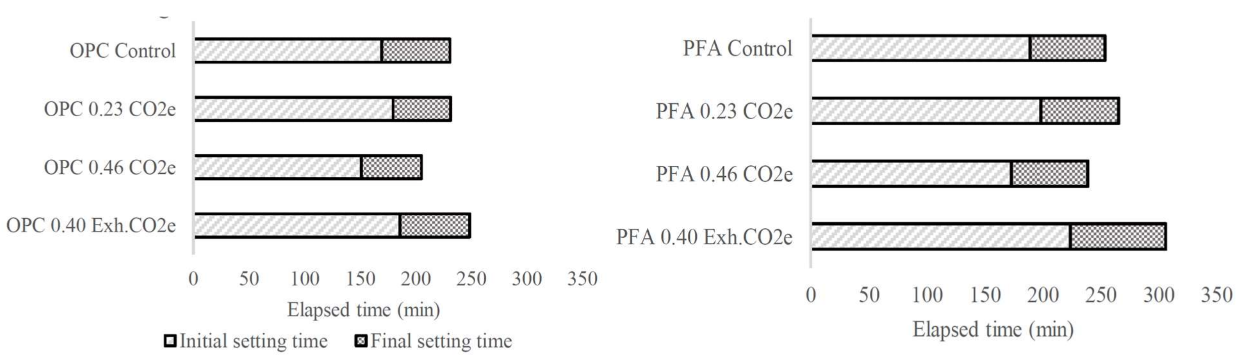


Figure 2. 4 Setting time of OPC and PFA concrete specimens (Samniang et al. 2021)

2.2.2 Mechanical Properties

Mechanical properties are the most important properties of concrete. These properties give an idea about the durability properties of concrete. The mechanical properties of nominal concrete and concrete added with carbon dioxide needs to be studied. Various studies carried out by researchers to understand the effect of the addition of carbon dioxide mechanical properties of concrete are discussed below.

(Sean Monkman and MacDonald 2017)found that the reduction in binder loading contributed to the reduction in compressive strength but the optimized dose of CO_2 while batching and mixing boosted its performance and improved the compressive strength. It is confirmed from 3-way comparisons that removing 7 to 8% of binder from a concrete mix would lead to a reduction in strength but the addition of CO_2 has the potential to restore the compressive strength performance.

(Sean Monkman et al. 2016)experimentally found that in CO_2 injected batches of dosage 0.05%, 0.15% and 0.30%, the best compressive strength was achieved by the lowest dose batch (0.05%) which provided a 14% improvement at 1 day and 10% at 3 days. The strength of concrete with the highest dose of CO_2 ranged from 5 to 11% which is lower than the reference. The range of doses used in different batches indicates that an optimal dose of CO_2 for strength development would be lower than 30% and likely in order of 0.05% to 0.15%.

(Kamal et al. 2020)proved that carbon dioxide sequestration increases the compressive strength of the concrete. Carbon dioxide sequestered concrete was found to have a higher compressive strength compared to the concrete without carbon dioxide because of the formation of nanosized calcium carbonate to fill the voids in the carbon dioxide sequestered concrete.

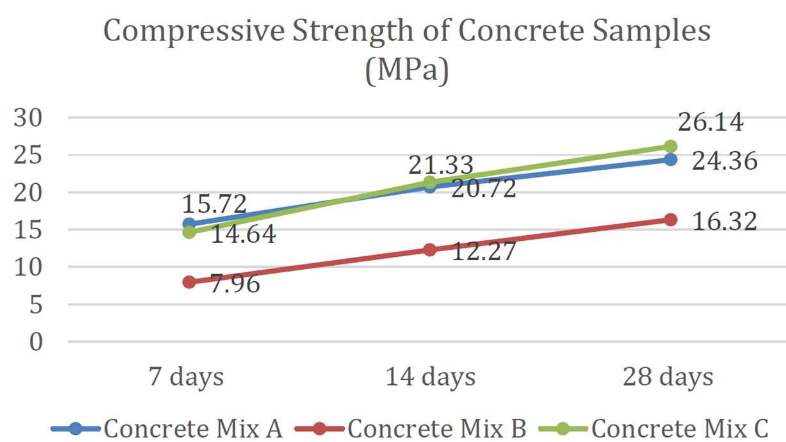


Figure 2. 5 Compressive strength of concrete samples on 7, 14 and 28 days (Kamal et al. 2020)

(S Monkman, MacDonald, and Hooton 2015)studied the effect of CO_2 injection into the concrete, it was evident from compressive strength results that there is a strength benefit of 20% at 3 days, 16% at 28 days and 26% at 58 days by addition of optimized dosage of CO_2. The strength was 26% better than control at 3 days, 15% better at 28 days and 18% better at 56 days.

(Sean Monkman 2018)found that at the optimal dosage of 0.11% by weight of cement/binder, provided the compressive strength performance consistent despite having 4.3% less binder (including 5.7% less cement).

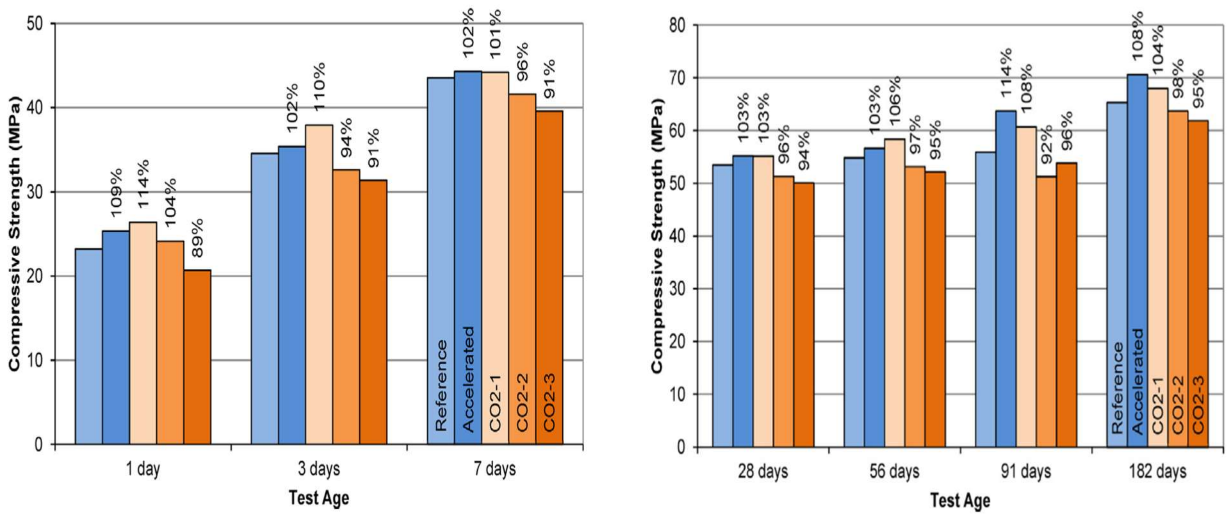


Figure 2. 6 Compressive strengths at 1, 3, 7, 28, 56, 91 and 182 days (Sean Monkman 2018)

(Samniang et al. 2021)prepared the mixture with and without fly ash replacement and then added pure CO_2 or exhaust gases (released from vans) in it. It was found that for mortar and concrete (without fly ash), the compressive strength of specimens with higher dosage of CO_2 slightly dropped except for the carbonated concretes mixed with $0.23CO_{2e}$. While for PPA concrete (with fly ash), the compressive strength of carbonated concretes (PFA 0.10 Exh CO_{2e} and PFA 0.20 Exh CO_{2e}) is higher than the controls.

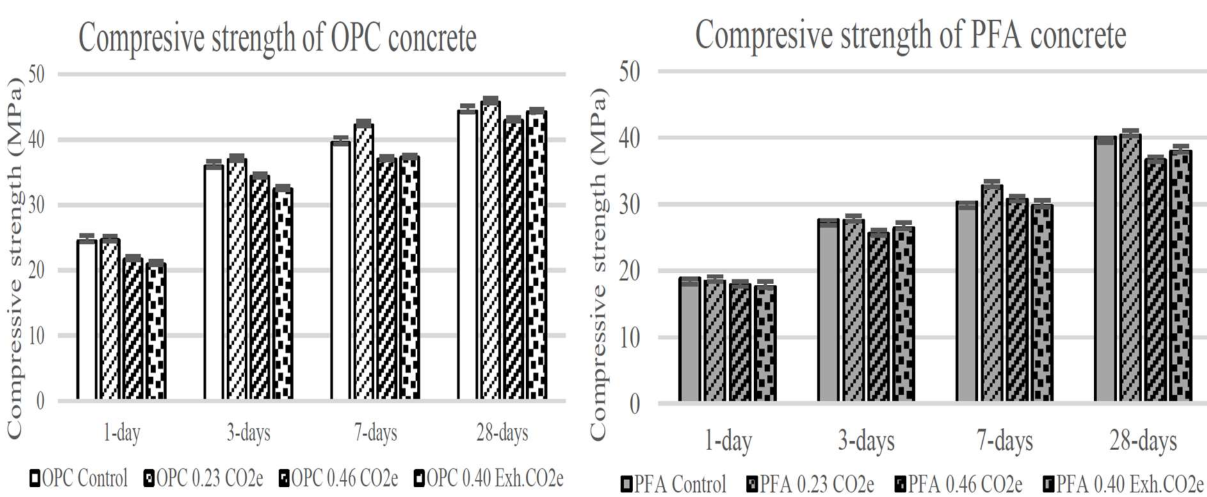


Figure 2. 7 Compressive strength of OPC and PFA concrete (Samniang et al. 2021)

(Tagwale et al. 2015)held a comparative experimental study of water cured concrete cubes and CO_2 cured concrete cubes for penetration and compressive strength and results indicated that CO_2 cured concrete cubes showed 22.125% higher compressive strength than water cured concrete cubes and CO_2 penetration of 13.5 mm after 2 hours.

(Y. Wang, He, and Yang 2018)examined the effect of dry ice on the hydration and hardening behaviour of Portland cement and found that the 7 days compressive strength firstly increases and then decreases with the increase in dry ice dosage. It was maximum at 0.6 wt% dosage with increase of 7.2% compared to control mix. The 7 days compressive strength decreased when dosage exceeded 0.9 wt%. The 28 days compressive strength showed the similar trend as that of 7 days, first increases and then decreases. The 28 days compressive strength was maximum at dosage of 0.6 wt% with increase of 30.9% compared to control mix. The 28 days compressive strength was higher than the control mix at the dosage of 1.5wt%.

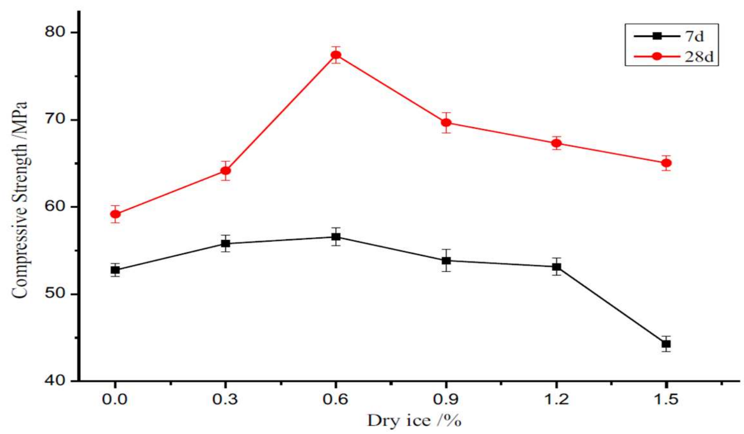


Figure 2. 8 Effect of dry ice on the compressive strength (Y. Wang, He, and Yang 2018)

(Santhosh Kumar et al. 2019)carried out the experimental studies on CO_2 cured, dry ice cured and water cured specimens for compressive strength, split tensile strength, and flexural strength and found out that CO_2 and dry ice cured specimens achieved higher compressive strength at 8 hours curing than water cured samples, and the highest strength was achieved by dry ice cured specimens.

(Qian et al. 2018)used CO_2 as an admixture through a technique called pre-carbonation method where CO_2 is first absorbed into a slurry of calcium-rich cementitious material and then blended with the rest of the ingredients to produce concrete. Experimental results confirmed that both compressive and flexural strengths of the OPC-based mortar samples were enhanced.

(S. Monkman and MacDonald 2016)produced the concrete blocks by adding carbon dioxide gas to the concrete during its mixing and forming stages. The carbon dioxide was absorbed into the concrete with the efficiency of 88%. The carbonation process showed the increase in compressive strength (13 to 33% at ages from 7 to 56 days) and decrease in absorption (by 18 to 36%).

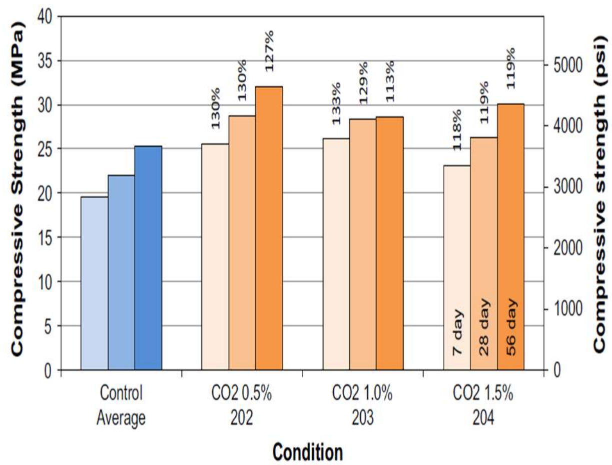


Figure 2. 9 Compressive strength at 7, 28 and 56 days of the mixes that included a water adjustment (S. Monkman and MacDonald 2016)

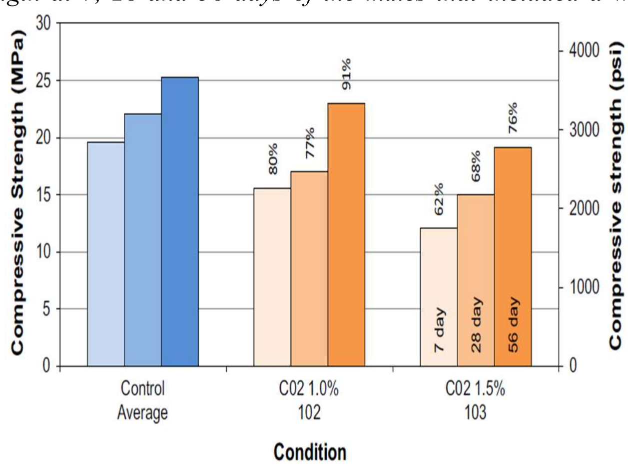


Figure 2. 10 Compressive strength at 7, 28 and 56 days of the mixes without a water adjustment (S. Monkman and MacDonald 2016)

(Rostami et al. 2012)examined the microstructure of OPC paste at early age carbonation curing and its effect on performance of cement paste at different ages. It was concluded that early carbonation followed by hydration accelerated the early strength by creating a microstructure with more strength contributing solids than normal hydration.

(Jang and Lee 2016)investigated the effect of the belite content and carbonation on the properties of cement mortar and found that carbonated belite rich Portland cement showed improvement in mechanical strength than normal cured for the same duration.

(D. Zhang, Li, and Ellis 2018)experimentally found that the extension of pre-hydration decreases CO_2 uptake and pre-hydration beyond the late deceleration period of Portland cement hydration (where heat generation is slowing down) leads to a higher hydrate content of cement paste, hence forming a higher compressive strength of mortar compared to the non-carbonated benchmarks at 28 days.

(Xuan, Zhan, and Poon 2016)used CO_2 gas pressure for mineral carbonation of concrete and found that curing for initial 6 hours had a positive influence on strength but became insignificant when extending it for 24 hours.

(Pan et al. 2017)attempted to extend the content of carbonate compounds in demolition RFA's by calcium hydroxide (CH) pre-soaking for escalating the carbonation effectiveness. The optimal curing conditions was found as 0.01-0.05 mol/kg CH solution, the CO_2 concentration of 7% and moisture content of 5%. After curing, it was found that powder content in RFA's got reduced to 9.1% from 14.2%. But there seemed to be an increase in compressive strength ratio from 0.95 to 1.04.

(P. He et al. 2016)examined the effects of additional water curing on the compressive strength and microstructure of CO_2-cured concrete. It was concluded that after CO_2 curing, a layer of $CaCO_3$ coating formed around the cement particles. The unreacted cement particles hydrated throughout the water curing process, and the concrete's compressive strength progressively enhanced. Concrete samples with a residual w/c ratio of 0.25 showed the greatest gain in strength. According to the results of mercury intrusion porosimetry tests, the porosity and pore size of the CO_2 cured mortar fell even more following water curing.

(B. J. Zhan et al. 2016) Implemented a CO_2 curing procedure to improve rapid strength development of concrete blocks using recycled aggregates. It was found that 2-hours of ACC resulted in an increase in compressive strength and curing degree. In terms of CO_2 diffusion, dissolution, and carbonation processes, a pressure increase of 0.1-0.5 bar produced superior results. In addition, it was advantageous in terms of compressive strength and degree of curing. Further water curing resulted in an increase in compressive strength, which is proportional to the early CO_2 curing degree. When cured with CO_2, concrete constructed of RA has superior fire resistance than standard procedures.

(T. Wang et al. 2017) Examined the effect of pressure, temperature and w/b on the CO_2 uptake during the MC curing. Pressure ranging from (0.5-2.5) is demonstrated to be most effective and desired for the process. After the low-ratio replacement of synthetic CS, the reaction rates were found to be greatly increased, as well as the CO_2 uptake capacity. The compressive performance of mixing CS specimens showed considerable enhancement following the MC curing.

(Shi et al. 2017) Utilized the accelerated carbonation for fast curing of concrete blocks. It emerged that the primary silicate phases (C_3S andC_2S) from stable calcium carbonates undergo a rapid chemical reaction, with CO_2 absorbing in the order $C_3S>C_2S>C_3A>C_4AF$. The water-cement ratio of 0.35-0.6 is considered. Before carbonation, the conditions for preconditioning must be met (Temperature = 22 ± 3°C, Relative Humidity = 55 ± 10%). Strengthening is facilitated by additional water curing.

(Ahmad et al. 2017) Presented experimental research on the effects of carbonation pressure and time on CO_2 uptake and strength evolution in a concrete mixture. It was found that ACC causes the compressive strength of concrete to improve over time, but at a slower rate. In terms of strength growth and CO_2 uptake, carbonation curing at a pressure of 60 psi (414 kPa) for 10 hours was determined to be the most effective. The examined concrete mixture's post-ACC compressive strength was more than 200 percent of its pre-ACC value, whereas CO_2 uptake was 11 percent.

(Xuan, Zhan, and Poon 2017b) Presented an experimental investigation to create a maturity technique for predicting the strength development of carbonated concrete blocks. It transpired that to achieve maturity index and strength growth, more CO_2 absorption with a rapid rate of gas flow and normal relative humidity is required.

(D. Zhang and Shao 2018) Presented a solution by using CO_2 curing to minimize the concrete surface salt scaling caused by freeze-thaw exposure. It has been observed that the early-age strength of concrete is improved by CO_2 curing, and the long-term strength is comparable to the non-carbonated control. In both concrete and pastes, CO_2 curing reduces the number of capillary pores exposed to water, allowing less water to freeze. The porosity of IPC was reduced by 40%, whereas it was reduced by 26% with FA pastes. The melting temperature of the material decreases significantly as the pore size is filled. As a result, a smaller percentage of pores freeze, reducing the damage to ACC concrete.

(Sharma and Goyal 2018) Examined the effect of carbonation curing on mortar mixes including cement kiln dust (CKD) as a partial replacement for cement. It was revealed that the compressive strength of mixtures containing CKD is increased by accelerated carbonation curing. Early compressive strength could be increased by up to 30% using ACC. When no further rehydration was done, carbonation curing yielded slightly lower 7-day and 28-day strength. The carbonated mortar specimen had a higher compressive strength than the water cured specimen after being sprayed with water following carbonation. Because of the densification of the mix, the permeability porosity of the mortars decreased after carbonation curing. SEM scans revealed the formation of calcium carbonate and a C-S-H gel. Even after the rapid carbonation curing method, the pH of the mortar sample remains in the high alkaline range.

(Ruonan et al. 2019) Investigated the carbonation curing of certain lightweight aerated concrete including fly ash, blast furnace slag, and red mud to substitute a portion of cement and lower the products' carbon footprint. The maximum CO_2 uptake was found in aerated concrete with red mud, followed by fly ash, and finally blast furnace. After four hours of CO_2 curing, aerated concrete containing 50% red mud had the maximum CO_2 absorption capacity (21.9 wt%), followed by fly ash (17 wt%), and finally blast furnace slag (15 wt%). During the first 20 minutes, approximately 80% of CO_2 uptake was achieved. The compressive strength of the blast furnace was 7.3 MPA, which was almost 197 percent higher than natural curing for the same time period.

(Chen and Gao 2019) Investigated the effects of pre-curing and carbonation time on the compressive strength and microstructure of Portland cement paste. Experimental results demonstrated that a water loss of 30–40% in cement paste is excellent for CO_2 uptake. The use of a combination of proper pre-curing and carbonation duration can effectively raise the compressive strength of cement mortar, especially at an early age, and it declines as the carbonation period is extended. Furthermore, when the carbonation curing period grows, the optimum pre-curing duration decreases.

(Z. He et al. 2019) Studied CO_2 sequestration in cement-bonded cellulose fiberboards using carbonation curing. It was observed that the highest feasible CO_2 uptake by conventional Portland cement with 18 h initial hydration, can reach 23.2 percent by 2 h carbonation and 28.5 percent by 24h carbonation. Cement-bonded cellulose fiberboards displayed excellent CO_2 absorption behaviour. The introduction of cellulose fibers improved the carbonate precipitation in cement matrix, with probable CO_2 uptake of 24.4 percent based on cement mass. The flexural strength obtained by carbonation curing can reach 70 to 100 percent of ultimate strength within 24 h. Carbonation curing can efficiently replace autoclave curing to accelerate strength.

(Meng et al. 2019) Investigated the impact of CO_2 curing method on the performance of cement blocks at high temperatures. It was evident that the CO_2 curing procedure could speed up the hydration cycle while also controlling mass loss. At increasing temperatures, the mass loss rates of CO_2 cured cement blocks were somewhat higher than the corresponding natural air cured blocks. The CO_2 cured blocks had a higher residual compressive strength than the corresponding natural air cured blocks, owing to a higher degree of cement hydration and calcium carbonate production.

(Ahmad et al. 2019) Investigated the effects of accelerated carbonation curing (ACC) on the performance of self-compacting concrete (SCC) mixtures made with limestone powder (LSP) alone or a combination of limestone powder and silica fume (SF) as mineral fillers. It was observed that after ACC for 10 hours, SCC combinations of limestone powder cement and silica fume cement showed a 68 percent and 42 percent improvement in compressive strength, respectively, compared to compressive values determined by testing at 18 hours after casting. Compressive strengths of around 95 percent and 85 percent of ultimate strength were obtained after ACC treatment for 10 hours, followed by 14 days of air curing, and moist-curing for 7 days followed by 7 days of air curing. For self-compacting concrete, the average depth of carbonation treated to ACC was 1.5 mm, indicating a lower risk of steel bar corrosion.

(Chen and Gao 2020) Investigated the effect of carbonation curing on the mechanical and durability performance of pervious concrete. It was concluded that carbonation curing for the right amount of time could improve the compressive strength of pervious concrete, especially in young specimens. Furthermore, the carbonation depth and degree of pervious concrete's interior layers are substantially higher than that of ordinary concrete. Pervious concrete, on the other hand, is more sensitive to carbonation curing duration than conventional concrete because of its lower cement content and higher carbonation depth.

(Qin and Gao 2019) Performed Carbonation curing on cement pastes containing varying amounts of waste autoclaved aerated concrete powder (0–50%). It was found that when cured with CO_2, the strength loss caused by the inclusion of waste autoclaved aerated concrete in Portland cement is compensated. In terms of compressive strength, 10-20% replacement yielded the best results.

2.2.3 Durability

Durability is the capability of concrete to withstand weathering action, chemical attack and abrasion without losing its engineering properties. The lesser volume of pores in concrete tend to make it more durable as it would help in reduction of intrusion of air, water or chemical to affect the bonding of aggregates. Different durability studies undertaken by researchers related to the addition of carbon dioxide in concrete are discussed below.

(Chen and Gao 2020) Investigated the effect of carbonation curing on the mechanical and durability performance of pervious concrete. Carbonation curing can increase freeze thaw durability to some level, based on relative dynamic modulus and mass loss with freeze thaw cycles, and the 6 h carbonation performs the best. Leaching and freeze-thaw cycling both enhance the porosity of cement paste in pervious concrete, however carbonation curing can counteract this effect.

(Z. He et al. 2019) Studied CO_2 sequestration in cement-bonded cellulose fiberboards using carbonation curing. It was found that carbonated fiberboards are more resistant to freeze-thaw cycling and wet-dry cycling due to the strengthened cement matrix by carbonate precipitation.

(Qin and Gao 2019) Performed Carbonation curing on cement pastes containing varying amounts of waste autoclaved aerated concrete powder (0–50%). It was found that the accelerated carbonation curing improves the resistance of PC-WAAC specimens to chloride ion penetration. PCWAAC blends containing 10–50 percent WAAC can successfully recycle 20 percent of WAAC to replace PC without sacrificing strength or chloride ion permeability, and PCWAAC blends including 10–50 percent WAAC can capture roughly 11.23–19.02 percent of CO_2.

(Mehdipour et al. 2019)studied the influence of pore saturation and CO_2 diffusivity on the carbonation and strength of OPC mortars. It was found that both traditional cement hydration and carbonation have similar levels of strengthening. And reducing pore saturation enhances carbonation if $S_{w,c} > 0.10$, otherwise carbonation is reduced/suppressed. Dry cast composites due to their lower water content and reduced surface coverage are more effectively carbonated.

(Gonen and Yazicioglu 2007)studied the influence of compaction pores on sorptivity and carbonation of concrete. It was concluded that the depths of carbonation were significantly affected by both the number of compacted pores and the relative humidity. Also, the rate of carbonation decreased with time. The maximum carbonation and sorptivity coefficient were found in non-compacted specimens while the minimum carbonation and sorptivity coefficient were found in specimens compacted by vibration. It was also found that change in the compaction pores affects the carbonation rate and the sorptivity coefficient.

(Younsi et al. 2011)found that whatever the more of curing, the designed mixture was as resistant to accelerated carbonation as the reference mixture containing blended cement. They also found that the tested mixture was less porous and more resistant to carbonation upon water curing than when air cured.

(Sharma and Goyal 2022)studied the near-surface properties of concrete subjected to accelerated carbonation curing. After 2-hours of preconditioning, freshly cast concrete was exposed to ACC for 6- hours, followed by water spray for 3-days. It was found that in carbonation cured concrete, water absorption, sorptivity, chloride permeability, and carbonation depth all decreased, indicating a drop in concrete surface permeability.

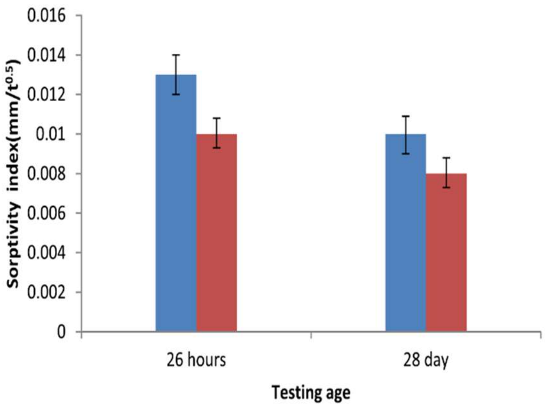


Figure 2. 11 Sorptivity coefficients for WC and CC concrete (Sharma and Goyal 2020)

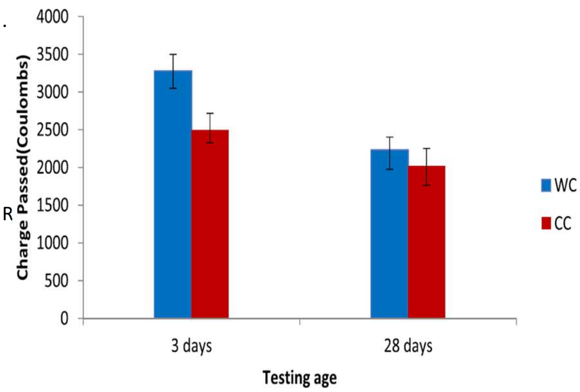


Figure 2. 12 Rapid chloride permeability test for WC and CC concrete (Sharma and Goyal 2020)

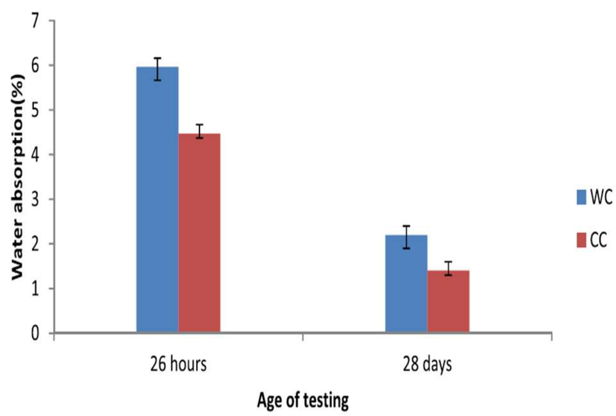


Figure 2. 13 Water absorption for WC and CC concrete (Sharma and Goyal 2020)

(Chun, Naik, and Kraus 2007)studied the effects of three different curing environments like most curing room, drying room, and CO_2 chamber on properties of concrete made by class C fly ash containing 0%, 18%, and 35% cementitious material. The results showed that the rate of carbonation of concrete was the highest in the CO_2 chamber, the abrasion resistance of carbonated specimens was lower than moist cured specimens, and specimens cured in the drying room showed the lowest strength and abrasion resistance.

(Sean Monkman et al. 2016)prepared five concrete mixtures in which three mixtures were treated with increasing dose of CO_2. All batches were found to have linear shrinkage lower than the optimal limit (0.04%), although the concrete with the highest CO_2 dose did show a small increase in linear shrinkage. The hardened air content and air void characteristics were below the maximum air void spacing factor limit of 0.230 mm for all the batches and were acceptable. The air content both in fresh and hardened states of mixture with CO_2 dosage 0.05% was lower than the reference. The RCPT test, bulk resistivity, rapid chloride migration test indicated that the CO_2 injection did not negatively impact the predicted transport properties of concrete. The non-steady state rapid chloride migration test indicated that all the CO_2 injected mixtures had lower chloride migration values than the reference mixture at 28 days. The mixtures with CO_2 injected dosage exhibited lower scaling than the mixtures without CO_2. The mixtures with lowest dosage of CO_2 (that is 0.05%) exhibited the least scaling with a 40% reduction over the non-CO2 mixtures.

Table 2. 2 Charge passed (coulombs) in the Rapid Chloride Permeability Test (RCPT) (Sean Monkman et al. 2016)

Batch	**28 days**	**56 days**	**180 days**
Reference	1563	1061	841
Accelerated	1653	1385	906
CO_2-1	1433	1126	965
CO_2-2	1597	1161	900
CO_2-3	1507	1114	836

Table 2. 3 Bulk resistivity test results (kΩ -cm) (Sean Monkman et al. 2016)

Batch	**28 days**	**56 days**
Reference	10.0	12.9
Accelerated	10.3	13.4
CO_2-1	9.9	13.3
CO_2-2	9.6	12.6
CO_2-3	10.1	13.0

Table 2. 4 Rapid chloride migration (NT 492) test results (10^{-12} m^2/s) (Sean Monkman et al. 2016)

Batch	**28 days**	**56 days**
Reference	8.2	6.3
Accelerated	5.6	5.8
CO_2-1	7.0	6.7
CO_2-2	7.1	6.0
CO_2-3	6.4	4.8

(Šavija and Luković 2016)reviewed the state of art regarding the understanding and consequences of carbonation of cement paste which has been recognized as one of the causes of reinforcement corrosion. Carbonation is sometimes used on purpose to improve the certain properties like strength, porosity, pore size distribution etc. of cement paste and can be used during accelerated curing for improvement of fiber reinforced cementitious composites, concrete recycling and waste immobilization.

(D. Zhang and Shao 2016b)examined whether early carbonation curing accelerates the chloride penetration and the weathering carbonation depth in concrete during its service. It was found that pH of concrete gets reduced immediately after carbonation and with more hydration for 27 days gets restored and are comparable to hydration reference. Fly ash OPC concrete showed chloride content less than 50% compared to the hydration reference. Thus, corrosion risk of reinforcing steel is highly reduced in concrete subjected to carbonation curing

(Peethamparan et al. 2003)studied the carbonation of concrete incorporated with GGBS, fly ash, and silica fume and found that at a lower water binder ratio (0.3 - 0.6) and prolonged curing led to a slower rate of carbonation. To improve carbonation resistance, sufficient water curing is done for plain concrete while curing is extended in case of concrete having GGBS, fly ash, and silica fume. Carbonation rate of low fineness GGBS (4500 cm^2 /g) and fly ash concrete increase with increase in OPC replacement. In case of silica fume 5% and 10% replacement led to lower carbonation rate. For concrete having GGBS of higher fineness (6000 - 8000 cm^2/g) and silica fume showed low carbonation rates than plain concrete. The carbonation coefficient increased with decrease in compressive strength linearly.

(D. Zhang and Shao 2016a)studied the unique process for early age carbonation curing of precast reinforced concrete to improve its performance and carbon storage capacity. It was found that 12-hour carbonation results in carbon uptake of 16% and reduces the pH of concrete surface to 9.2 while the pH of 13 is maintained at the core. The subsequent hydration restores the pH of the surface to 12.3 compared to hydration reference.

2.2.4 Microstructure

Micro structural analysis of concrete is a novel approach for determining the morphological properties of concrete. The general techniques used to investigate the micro structural behaviour of concrete include X-Ray Diffraction Analysis (XRD), Scanning Electron Microscope (SEM), and Energy Dispersive Spectroscopy (EDS). These advanced tools may examine the unique features of the concrete. The mineral data gathered from the micro structural analysis will aid in interpreting *the distinctive properties of concrete as well as the existence of small compounds within hardened concrete. The effect of the addition of carbon dioxide on the microstructure of concrete is discussed below.*

(Shah et al. 2018)studied the influence of carbonation on the microstructure of cement pastes with OPC, PPC, and LC^3 using XRD, TGA, SEM, and MIP. It was found through XRD that all the hydrated phases get carbonated simultaneously upon exposure to CO_2, the total amount of amorphous content in carbonated samples was lower as compared to uncarbonated samples indicating carbonation of amorphous calcium bearing phases that is CSH. According to the TGA results, lower mass loss due to the decomposition of calcium carbonate was observed in naturally carbonated OPC and PPC samples as compared to accelerated carbonation, implying a lower degree of carbonation in the naturally exposed samples**.** The total MIP was lowest in the OPC system. Also, the increase in porosity was observed in the PPC and LC^3 system on carbonation, because it contains a lower amount of CH than OPC resulting in extensive carbonation of CSH and other hydration phases which results in a reduction in the total solid volume. In SEM Images, the clear distinction was observed between carbonated and uncarbonated samples. The uncarbonated samples had at distinct contrast between different phases in the system, the brightest regions corresponded to anhydrous cement grains; light grey region corresponded to calcium hydroxide and CSH and black colour represented pores. While in carbonated samples the contrast was reduced and throughout the image brighter products seemed to appear which corresponded to polymorphs of calcium carbonate precipitated on carbonation intermixed with decalcified CSH or silica gel.

(Cui et al. 2015)analysed the diffusion mechanisms of CO_2 gas in concrete through the SEM observations. It was observed that the concrete samples with high CO_2 concentrations have formed a dense carbonated concrete microstructure at its outermost layer and thus reducing the rate of CO_2 diffusion. The SEM images also showed that the uncarbonated G30 samples were amorphous, fibroid and lumpy while the carbonated G30 samples had high porosity with large interconnected pores. The MIP method revealed that the porosity of uncarbonated concrete (G30) at 7 days was found to be 11.2% while the porosity of carbonated concrete (G30) at 10% CO_2 concentrations was reduced to 9.8%, the porosity of carbonated concrete (G30) at 20% CO_2 concentrations were further reduced to 8.7% and the porosity of carbonated concrete (G30) at 50% CO_2 concentrations were lowest at 8.1%.

(Samniang et al. 2021)prepared the mixture with and without fly ash replacement and then added pure CO_2 or exhaust gases (released from vans) in it. Thermogravimetric analysis revealed that for mortar and concrete (without fly ash), on comparing the control mix with the concrete mix with CO_2 added, with the increasing dosage of CO_2, the $Ca(OH)_2$ content reduces while the $CaCO_3$ content gradually increases. While for PFA concrete (with fly ash), the higher $CaCO_3$ content was detected in the mixtures injected by CO_2 (PFA 0.10 Exh CO_{2e} & PFA 0.20 Exh CO_{2e}).

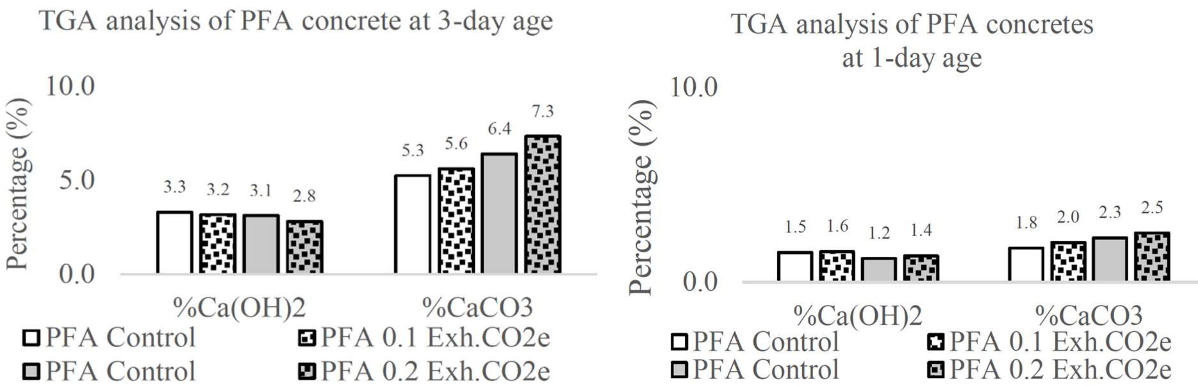


Figure 2. 14 TGA analysis of PFA concretes at 1- and 3-day age (Samniang et al. 2021)

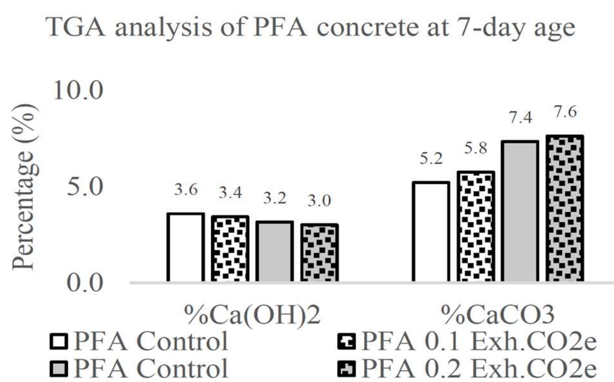


Figure 2. 15 TGA analysis of PFA concretes at 7-day age (Samniang et al. 2021)

(Chen and Gao 2019) Investigated the effects of pre-curing and carbonation time on the compressive strength and microstructure of Portland cement paste. According to MIP data, both hydration and carbonation reduced the porosity of cement paste. The large capillary holes are reduced by carbonation, but the small capillary pores are reduced by water curing. It is necessary to do adequate pre-curing and CC; otherwise, de-calcification of C-S-H would occur, resulting in a loss of strength.

(Meng et al. 2019) Investigated the impact of CO_2 curing method on the performance of cement blocks at high temperatures. SEM analysis shows that CC has positive outcomes because it alters and fills the microstructure of cement at a higher temperature of 600C. The process of CO_2 curing facilitates the consumption of $Ca(OH)_2$ to create $CaCO_3$, which affects the mechanical and physical properties, according to DTG and XRD analyses.

(Y. Wang, He, and Yang 2018)examined the effect of dry ice on the hydration and hardening behaviour of Portland cement. The results of hydration heat analysis, XRD, SEM and DTA-TG show that with the addition of dry ice, the early hydration was delayed (retarded) but improved its later hydration and optimized the hardened structure of the cement paste.

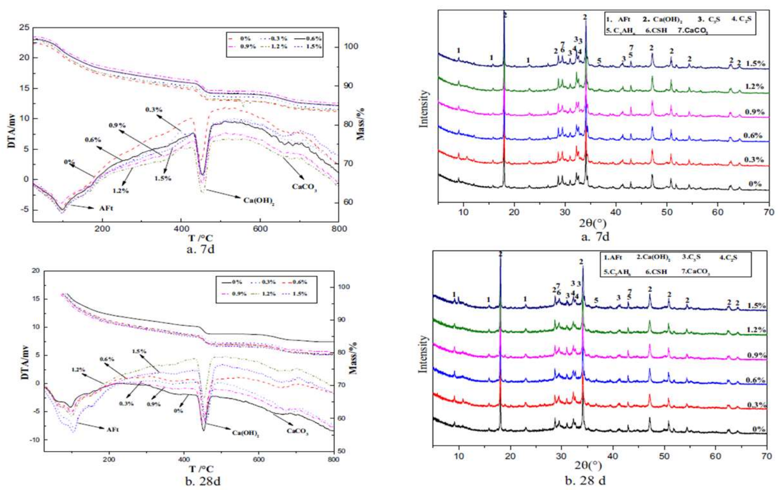


Figure 2. 16 DTA-TG diagram and XRD patterns of samples at 7 and 28 days (Y. Wang, He, and Yang 2018)

(Qian et al. 2018)used CO_2 as an admixture through a technique called pre-carbonation method where CO_2 is first absorbed into a slurry of calcium-rich cementitious material and then blended with the rest of the ingredients to produce concrete. SEM analysis revealed the formation of nano to sub-micro $CaCO_3$ particles which provides extra heterogeneous nucleation sites for hydration of OPC and densifies its microstructure which increases the strength of produced mortar/concrete.

(Mo and Panesar 2012)investigated the effect of accelerated carbonation curing on the paste with 0-40% reactive MgO. Outcomes revealed that the combined effect of carbonation and reactive MgO resulted in a reduction in pore size and total pore volume by 32%, increase in apparent density and 39% greater mean microhardness as compared to OPC pastes.

(Sharma and Goyal 2022) Studied the near-surface properties of concrete subjected to accelerated carbonation curing. After 2-hours of preconditioning, freshly cast concrete was exposed to ACC for 6- hours, followed by water spray for 3-days. In the near-surface of ACC concrete, TGA and XRD analysis revealed the existence of $CaCO_3$ and an increased quantity of CSH gel. SEM pictures revealed microstructure densification due to $CaCO_3$ precipitation in voids.

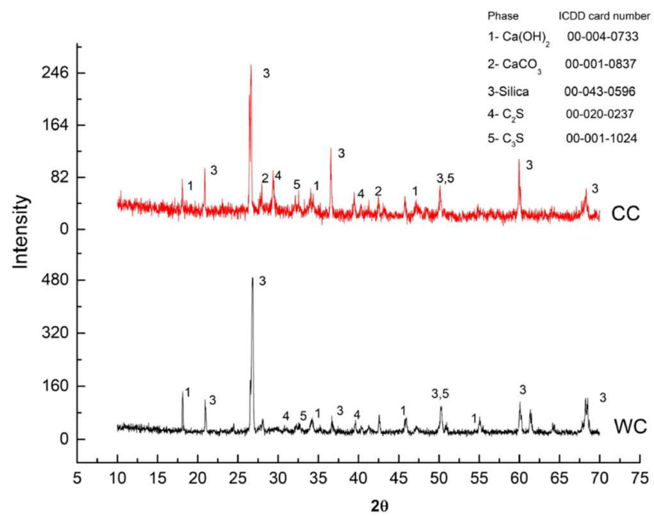


Figure 2. 17 XRD spectrum obtained for WC and CC concrete mixes at 28 days of casting (Sharma and Goyal 2020)

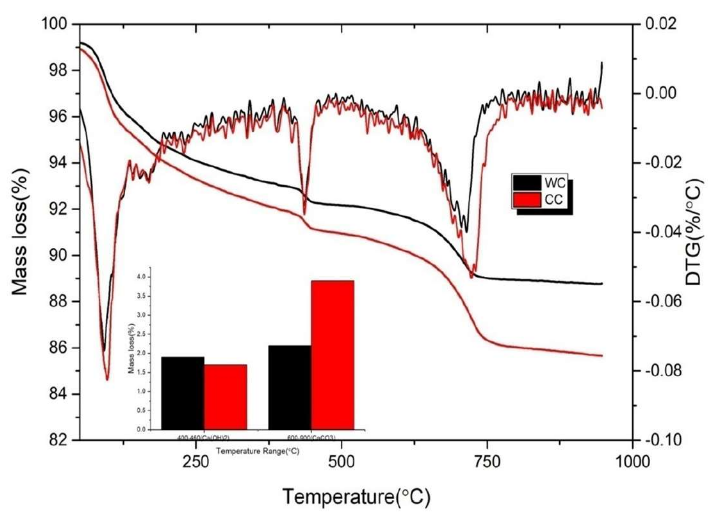


Figure 2. 18 TGA plot at the age of 28 days of casting (Sharma and Goyal 2020)

(Jang and Lee 2016) Investigated the effect of the belite content and carbonation on the properties of cement mortar. According to MIP results, the carbonated cement with high alite content showed increase in pore connectivity while with high belite content showed reduced pore connectivity, thus possessing complex microstructure. Densification of pores in the range of 50 mm to 10 micro m that influence the strength.

2.2.5 Environment

The environmental effect is comprised of a wide range of advantages that have an influence on the environment. Firstly, recycled CO_2 is permanently trapped in concrete. Carbon dioxide is the most well-known and principal greenhouse gas, accounting for the largest percentage of all greenhouse gases. As a result, concrete sequestration will significantly reduce CO_2 emissions in the environment, particularly from the construction industry.

(Shao, Monkman, and Boyd 2010) Commonly used precast concrete products such as masonry units, paving stones, cement boards and fiber boards are ideal for CO_2 sequestration. Being in high demand as their usage is more often, the cement consumed in their production is also massive. On CO_2 curing these products, it not only can sequester the massive amount of CO_2 but also can reduce cement usage without compromising on strength.

(Adesina 2020) Sustainability has led the concrete industry to reduce the CO_2 emissions which are mainly produced due to the production of Portland cement. For the past decade, several initiatives have been taken and will be taken to achieve net-zero emissions by 2050. Concrete continues to be the preferred material in the construction industry and it is required to meet future demands in an eco-friendly way. We have to control CO_2 emissions by replacing Portland cement partially or fully and by obtaining materials from local sources so thereby controlling CO_2 emissions and reducing the cost of concrete.

(Sean Monkman, Kline, and Cail 2019) The CO_2 utilization has been emphasized and demonstration was performed where novel Cryogenic technology was used to capture CO_2 and was injected in the concrete mix. This resulted in the formation of $CaCO_3$ that impacted concrete hydration. The motive is to demonstrate the viability of a complete supply chain solution so that all of CO_2 is consumed. Rather than relying on yet to be made fully integrated and full-scale capture and utilization of CO_2 from cement plants, the project demonstrates that a utilization scaled capture solution is capitally efficient. Time is an important factor here and as a fact, carbon reductions now are more valuable compared to the future.

(El-Hassan and Shao 2014) Investigated the impact of initial curing on the carbonation curing of lightweight concrete masonry units (CMU). With first pre-curing, it was discovered that CO_2 uptake capability is 22 percent to 24 percent. For 4-day carbonation, the absorption capacity is 35 percent, compared to 8.5 percent without first curing. To improve and efficiently recycle cement kiln CO_2, the CC can replace steam curing in concrete masonry unit manufacture.

2.2.6 Recycled Concrete Aggregates (RCA)

The recycled concrete aggregates produced from the demolished wastes of the buildings are weaker than the normal aggregates, due to the attached mortar with large number of pores or the cracks formed during crushing. Thus, in order to improve their properties, these are subjected to CO_2 curing. The CO_2 enhances its properties in such a way that its properties are almost similar to the natural aggregates.

(B. Zhan et al. 2014) Experimentally studied the properties of concrete prepared with recycled mortar aggregate (RMA) that has been modified by a CO_2 curing method. A total of five mixes were prepared; one control mix, two RMAs RMA1 & RMA2, and two CI-RMAs CI-RMA1 & CI-RMA2. Based on the results, it was concluded that the compressive strength and tensile splitting strength of the concrete made with RMA and CI-RMA were lower than that of the control concrete at 28 days. However, at 90 days, the compressive strength of concrete made with CI-RMA2 was similar to the control concrete while the tensile splitting strength of the concrete mixture made with RMA and CI-RMA was slightly higher than that of the control concrete. The drying shrinkage of the concrete mixtures made with CI-RMA1 and CI-RMA2 was approximately 10% and 15% lower than that of RMA1 and RMA2 respectively. The mix prepared with RMA2 had the highest slump value of 165mm. At 28 days, the concrete made with CI-RMA1 and CI-RMA2 had approximately 41% and 46% higher resistance to chloride penetration than that of concrete made with RMA1 & RMA2 respectively.

(Xuan, Zhan, and Poon 2017a) Studied the durability properties of RAC prepared with non-carbonated RCAs and carbonated RCAs. Two RCAs were prepared NRCA's and ORCA's. The experimental results demonstrated that by addition of carbonated RCAs to RAC reduced not only its water absorption but also its permeability. By the usage of 100% carbonated NRCA's, improved bulk electrical conductivity, chloride ion permeability and gas permeability by 15.1%, 36.4%, and 42.4% respectively. The drying shrinkage of RCAs with 100% non-carbonated NRCA's had the highest drying shrinkage. When the findings of mechanical and durability characteristics were compared, the CO_2 curing treatment of RCAs had a stronger favorable influence on the RAC's durability qualities. Also, the water absorption values of RAC may be used to determine the durability of RAC.

(B. Zhan, Poon, and Shi 2013) Adopted a CO_2 curing process to cure concrete blocks made with recycled aggregates. To replace natural aggregate, non-load-bearing and load-bearing blocks were made using 0%, 50%, and 100% recycled aggregate. The findings revealed that the CO_2 curing procedure is a novel way for creating recycled aggregate concrete blocks. The replacement ratio of recycled aggregate had no discernible effect on CO_2 curing degree, presumably due to the high-water content of blocks containing a high percentage of recycled aggregate interfering. CO_2 curing enhanced the compressive strength of both non-load-bearing and load-bearing blocks considerably and quickly when compared to standard wet curing, with strength improvements ranging from 108% to 151% in under 24 hours. Furthermore, CO_2 curing greatly decreased the drying shrinkage values of the blocks.

(J. Zhang et al. 2015a) Attempted to improve the quality of RCAs through carbonation of the attached cement paste. The test results concluded that carbonation enhanced the density and apparent density (4.4-5.2%) of the connected cement paste while decreasing water absorption (22.6-28.3) and crushing value (7.6-9.6%) of the RCAs. During carbonation, the solid volume increased due to the formation of both $CaCO_3$ and silica gel, which contributed to the decrease in absorption and rise in density of the RCAs. Fresh mortars containing carbonated RCA flowed somewhat better than those containing uncarbonated RCA, while mortars containing C-RCA flowed better than those containing G-RCA. The compressive strength of carbonated RCA mortars was greater than that of uncarbonated RCA mortars. After carbonation, the strength increase of mortar with G-RCA was more than that of C-RCA.

(Sereng et al. 2020) Studied the effectiveness of accelerated carbonation of recycled concrete aggregates to achieve the highest CO_2 uptake on RCA while also improving their characteristics. The findings reveal that with an optimal water content, a maximum CO_2 uptake may be reached, which is dependent on RCA characteristics. RCA-CB 1-4 have a higher CO_2 uptake of 49.9 g/kg due to their origin. CO_2 uptake can be improved by increasing CO_2 concentration until it reaches 100% CO_2, and fine RCA have a higher CO_2 uptake than coarse RCA (21.8 g/kg for RCA 1-4 mm vs. 8.5 g/kg for RCA 12-20 mm). Finally, CO_2 uptake has shown to have a considerable impact on the reduction of the water absorption coefficient from 36 to 43%.

(Silva et al. 2015) Studied the influence of integrating recycled aggregates from construction and demolition waste on the carbonation behaviour of concrete. It was found that carbonation depths increase as the amount of RA incorporated increases. The use of 100% coarse RCA in concrete can result in carbonation depths that are up to two times higher than those of similar NAC mixtures. RMA causes larger carbonation depths than RCA for a given replacement level. The carbonation behaviour of concrete is also affected by the size of the RA. Concrete mixes created with fine RCA are more likely to have larger carbonation depths than mixes made with coarse RCA. Using water reduction admixtures to lower the w/c ratio in RAC mixes is an excellent way to improve mechanical performance while also lowering carbonation. Mineral additives as a cement substitute result in larger carbonation depths than mixtures without them. When compared to NAC, RAC mixes have a comparable connection between the coefficient of accelerated carbonation and compressive strength.

(Zhao et al. 2018) Studied the possibility of improving the properties of RCA (non-carbonated RCA (RCAl nc), well-carbonated RCA (RCAl wc), and industrial RCA (RCAi) by accelerated carbonation. According to TGA data, the finer fraction of the RCA had a larger amount of portlandite, a higher hardened cement paste content, and a higher porosity than the coarser fraction. Due to the transition of portlandite into calcite, the density of all fractions of RCAl wc was greater than that of RCAl nc. Because of the reduction in hardened cement paste porosity owing to carbonation, the water absorptions obtained for all granular classes of RCAl wc were much lower than those obtained for RCAl nc, according to the MIP data.

(Singh et al. 2021) To increase the quality of recycled coarse concrete aggregates using the carbonation process, three mix combinations were created viz NAC (100% natural coarse aggregate), RAC (100% recycled coarse aggregate), and CRAC (100% carbonated recycled coarse aggregate). According to the test results, the UPV value of the 100% CRAC increases by 6.38% when compared to the 100% RAC, but decreases by 4.27% when compared to the 100% NAC. At 28 days, the compressive strength of the 100% CRAC increased by 21.05% when compared to the 100% RAC, but decreased by 8.63% when compared to the 100% NAC. At 28 days, the flexural strength of the 100% CRAC increased by 30.32% when compared to the 100% RAC, but decreased by 12.63% when compared to the 100% NAC. Split tensile strength followed a nearly identical pattern as that of flexural strength. After 28 days, the chloride-ion penetration of the 100% CRAC has decreased by 14.26% when compared to the 100% RAC, but has increased by 9.72% when compared to the 100% NAC. Water absorption and water penetration results were substantially superior when comparing CRAC to RAC than comparing RA to RAC. The sorptivity of the 100% CRAC decreases after 28 days when compared to the 100% RAC, but increases when compared to the 100% NAC, 100% RAC displayed the highest value.

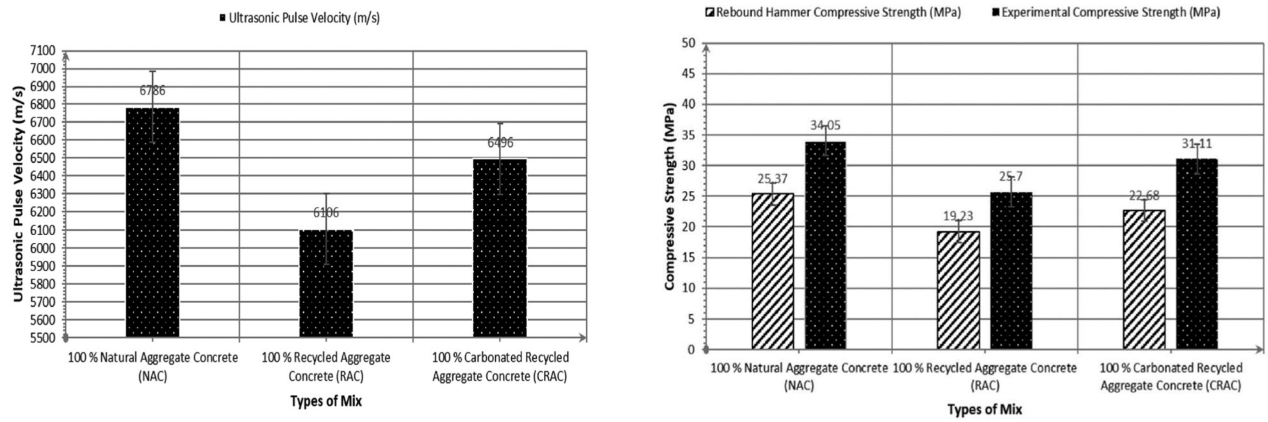


Figure 2. 19 Comparison of rebound hammer and experimental compressive strengths (left), and UPV values (right) for all types of mix at 28 days (Singh et al. 2021)

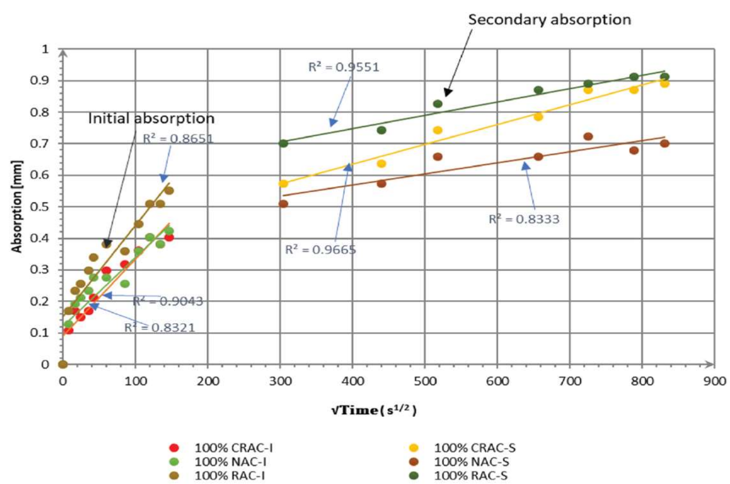


Figure 2. 20 Sorptivity test results for all types of the mix at 28 days (Singh et al. 2021)

(Ghouleh, Guthrie, and Shao 2017) Demonstrated the ability to make artificial carbonate aggregates out of steel slag that has been optimally activated by carbon dioxide for waste recycling and carbon usage in concrete. It was concluded that the granule's absorption capacity was boosted by 17% of their unit weight after they were double carbonated. After only two hours of carbonation, the dense pore microstructure was observed that resulted in superior transport properties and durability, and better resistance to ingress and leaching. Also, good resistance against the effect of freeze-thaw was observed.

(J. Zhang et al. 2015b) Investigated the effect of carbon dioxide treatment of recycled concrete aggregate (RCA) on the performance of RCA and RCA mortar. The results indicated that carbonation raised the apparent density (4.7-5.6%) and decreased both water absorption (7.6-9.6%) and the crushing value (22.6-28.3%) of the RCA. The RCA mortar has a lesser flowability and compressive strength than natural sand mortar. However, after CO_2 treatment, the characteristics were extremely close to natural sand mortar. The carbonated RCA mortar showed enhanced autogenous shrinkage, decreased drying shrinkage, water absorption, and chloride migration coefficient when compared to the un-carbonated RCA mortar. The interfacial transition zone (ITZ) in the RCA and RCA mortar was examined using a scanning electron microscope (SEM). It was discovered that carbonation treatment of the RCA enhanced not only the original ITZ in the RCA, but also the newly produced ITZ in the RCA mortar.

(B. Zhan et al. 2014) Studied the use of a carbonation process to enhance the properties of recycled aggregates. According to the findings of the experiment, the CO_2 curing method can densify the mortar bonded to the RCA. The RCA's water absorption and porosity were significantly reduced after the CO_2 curing process. RCA with smaller particle sizes carbonated more easily due to the large specific surface area. Because the dry matrix could not provide enough water for the carbonation processes, and the pores in the water saturated matrix were filled with water blocking CO_2 penetration, the moisture content of RCA had a major impact on the carbonation percentage. Furthermore, the carbonation process moved quickly for the first two hours but then slowed dramatically after that.

(Park et al. 2020) Employed the quantitative data from the batch and column tests to investigate the potential of using $scCO_2$-sparging for processing concrete waste to produce a recycled building resource. The carbonation of concrete aggregate was dramatically increased even when only 1 hour of $scCO_2$ sparging and 2 hours of stationing were used. Considerable amount of the portlandite in the recycled aggregates was consumed, according to XRD, SEM/EDS, and TG/DTA studies and calcite was formed as a secondary mineral in its place after only 3 hours of treatment (1 hour of $scCO_2$ sparging and 2 hours of stationing), showing that the aggregate was adequately carbonated. The average pH of $scCO_2$-sparged aggregate reduced from 12.0 to <9.8 according to the results of batch extraction studies for both recycled aggregate types. Also, the pH of the eluent from the column containing the $scCO_2$-sparged aggregate stayed at< 9.8, indicating that a one-hour $scCO_2$ sparging method is adequate to carbonate waste concrete aggregate and produce a new construction material resource.

(Pan et al. 2017) Attempted to extend the content of carbonate compounds in demolition RFA's by calcium hydroxide (CH) pre-soaking for escalating the carbonation effectiveness. The optimal curing conditions was found as 0.01-0.05 mol/kg CH solution, the CO_2 concentration of 7% and moisture content of 5%. After curing, it was found that powder content in RFA's got reduced to 9.1% from 14.2%. Also, there was a decrease in water absorption from 4.35% to 1.65%, crushing value from 18% to 13%, and water demand ratio from 1.17 to 1.10. But there seemed to be an increase in compressive strength ratio from 0.95 to 1.04.

(B. Zhan, Poon, and Shi 2016) Explored the influence of material properties on CO_2 curing of concrete blocks. It was transpired that after CO_2 curing, the apparent density increases from 2995 to 2222 kilos per meter cube due to the reduction in permeability. The amount of CO_2 produced is determined by how much RA is used. It compensates for the loss of strength caused by the RA's lower grade. Conditions that are very moist or dry are adverse to CC. Concrete blocks must be pre-treated to minimize their moisture content. When compared to steam curing, 2-hour CC produces the same quality product.

CHAPTER 3

Experimental Program

3.1 General

This chapter describes the materials used in the experimental program, the design compositions of the experimental samples, and the methodology adopted to conduct the experiments. The first section deals with the materials used to make the concrete and cement mortar mixes, their physical properties, and their chemical compositions. Concrete mix proportions, their design compositions, mixing procedures, and wet concrete properties are mentioned in the next section. The experimental setup, the tests adopted, and the reference codes involved in the testing process have been detailed inthe subsequent sections. The testing methodology has been classified into three categories; mechanical strength, durability, and microscopic.

3.2 Materials used in the study

The binder materials used in the present study are Ordinary Portland Cement (43 grade), conforming to (IS: 8112 – 1989), and Portland Pozzolana Cement, conforming to (IS:1489 (Part 1) 1991). Crushed basalt was used as coarse aggregate and sand as fine aggregate.

3.2.1 Cement

Cement is one of the best binding materials used in concrete. Two different types of cement, i.e., OPC and PPC fly ash-based cement (manufactured by UltraTech Cement Pvt. Ltd.) conforming to (IS: 8112 – 1989) and (IS:1489 (Part 1) 1991), respectively from a single lot, were used for preparing concrete mixes throughout this study. The cement samples were collected and tested according to the guidelines of (IS: 4031(Part11):1988), (IS: 4031 (Part 5) 1988), and(IS: 4031 (Part 6) 1988). The properties of the cement investigated were Specific-Gravity, initial setting time, consistency, etc. The physical properties of the cement are presented in Table 3.1.

Table 3. 1 Physical properties of the cement

Properties	OPC 43	PPC
Specific Gravity	3.15	3.06
Initial-Setting Time (min)	112	54
Consistency of Standard Cement paste (%)	28	34
Final-Setting Time (min)	281	205

3.2.2 Aggregates

Aggregates fill the maximum volume of the concrete and are assumed to have a high impact on the properties of concrete. The aggregates should be in proper shape and size, clean, hard, and well graded. Based on the size of the particle, the aggregates are divided into two groups

- **Fine aggregates:** The particle size is less than 4.75 mm and,
- **Coarse aggregates:** The size of the particle is more than 4.75 mm.

3.2.2.1 Fine aggregates

Locally procured river sand was used as fine aggregates. The particle size distribution (sieve analysis) and other physical properties of the fine aggregateare presented in Table 3.2 and Table 3.3, respectively. The sand was first sieved through a 4.75 mm sieve to remove any particles greater than 4.75 mm and then washed to remove the dust. The fine aggregates were tested as per Indian Standard Specifications (IS:383 1970). Based on particle size distribution, the fine aggregates fall under zone II of (IS:383 1970).

Table 3. 2 Sieve analysis of fine aggregates

I. S. Sieve	Weight retained(gm)	Percentage weight Retained	Cumulative % of weight Retained	Cumulative % of weight Passing	Limit by IS 383 for Zone-II
4.75 mm	13	1.3	1.3	98.7	**90 to 100**
2.36 mm	10	1	2.3	97.7	**75 to 100**
1.18 mm	385	38.5	40.8	59.2	**55 to 90**
600 µm	169	16.9	57.7	42.3	**35 to 59**
300 µm	267	26.7	84.4	15.6	**8 to 30**
150 µm	120	12	96.4	3.6	**0 to 10**
Pan	36	3.6	-	-	
Total	1000	100	282.9		
Fineness Modulus		**2.829**		**Hence, Zone-II**	

Table 3. 3 Physical properties of fine aggregates

Characteristics	Obtained Results	Requirements as per IS 383:1970
Grading	Zone-II	**Zone-II**
Fineness Modulus	2.829	**2.0 to 3.5**
Weight of saturated sand A (gm)	500	
Weight of pycnometer with sand B (gm)	1833	
Weight of pycnometer with Water C (gm)	1522	
Weight of Oven dry aggregate D (gm)	560	
Specific gravity = (A/ [A-(B-C)]) =	2.65	**2.6 to 2.7**
Weight of saturated sand A* (gm)	1000	
Weight of Oven dry sand B* (gm)	984	
Water Absorption = (B* - A*)/(A*)	**1.6%**	

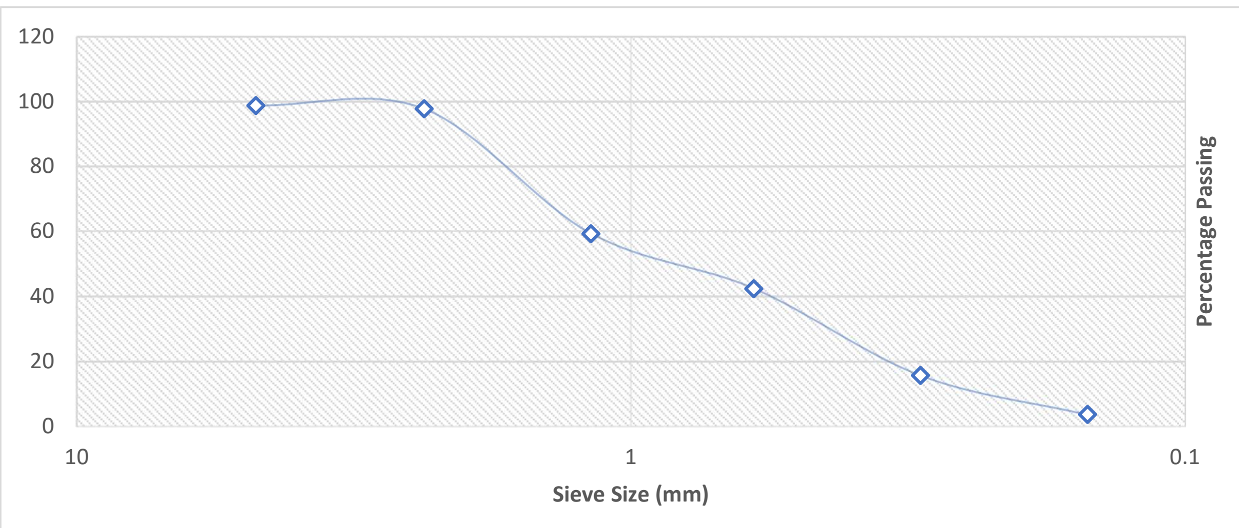


Figure 3. 1 Particle size distribution of fine aggregate

3.2.2.2 Coarse aggregates

Crushed stone aggregates (locally available) of 20 mm and 10 mm are used throughout the experimental study. The particle size distribution and physical properties of coarse aggregates are given in Table 3.4, Table 3.5, and Table 3.6.

Table 3. 4 Sieve analysis of 10 mm coarse aggregates

I. S. Sieve	Weight retained (gm)	Percentage weight Retained	Cumulative % of weight Retained	Cumulative % of weight Passing	Limit by IS 383
80 mm	0	0	0	100	**-**
40 mm	0	0	0	100	**-**
20 mm	0	0	0	100	**-**
12.5 mm	0	0	0	100	**100**
10 mm	813	16.26	16.26	83.74	**85 to 100**
4.75 mm	3643	72.86	89.12	10.88	**0 to 20**
2.36 mm	289	5.78	94.9	5.1	**0 to 5**
Pan	255	5.1	-	-	-
Total	5000	100	200.28		
Fineness Modulus =		$\frac{200.28 + 400}{100}$		= **6.0028**	

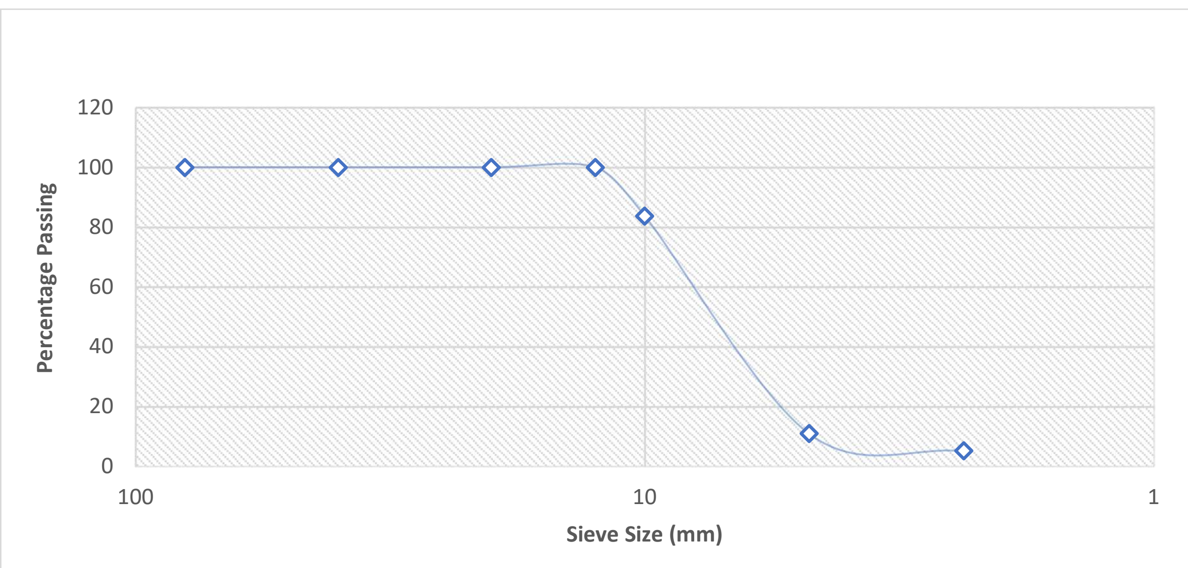


Figure 3. 2 Particle size distribution of 10mm coarse aggregate

Table 3. 5 Sieve analysis of 20mm coarse aggregates

I. S. Sieve	Weight retained (gm)	Percentage weight Retained	Cumulative % of weight Retained	Cumulative % of weight Passing	Limit by IS 383
80 mm	0	0	0	100	**-**
40 mm	0	0	0	100	**100**
20 mm	114	2.28	2.28	97.72	**85 to 100**
10 mm	4567	91.46	93.62	6.38	**0 to 20**
4.75 mm	301	6.02	99.64	0.36	**0 to 5**
2.36 mm	12	0.24	99.88	0.12	**-**
Pan	06	0.12	-	-	-
Total	5000	100	295.42		
Fineness Modulus =			$\frac{295.42+400}{100}$	**=6.9542**	

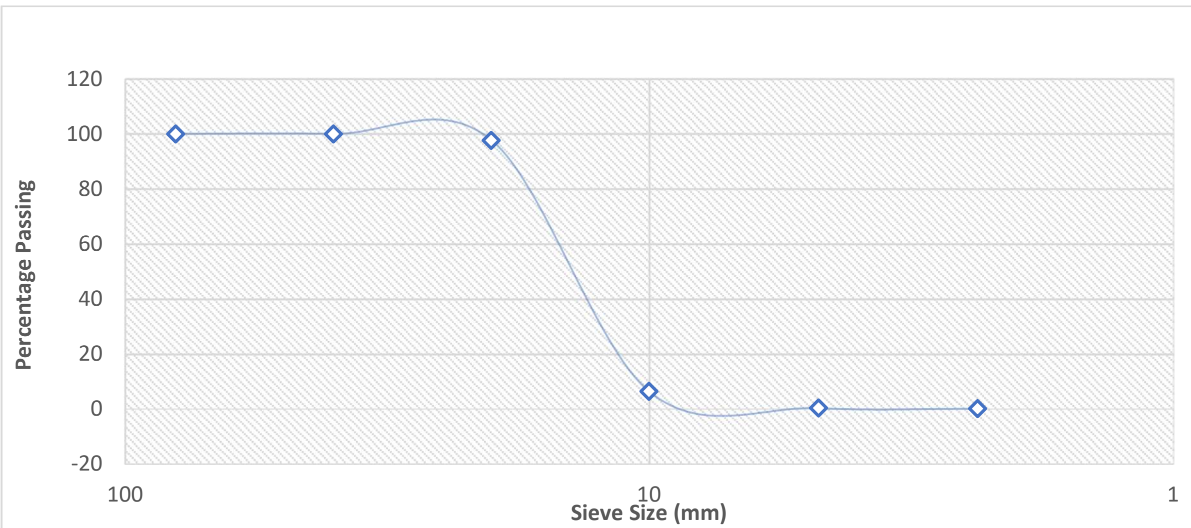


Figure 3. 3 Particle size distribution of 20mm coarse aggregate

Table 3. 6 Physical properties of coarse aggregates

Characteristics	Obtained Results for 10mm	Obtained Results for 20mm	Requirements per IS 383:1970
Fineness Modulus	6.003	6.954	**5.5 to 8.0**
Specific gravity	2.68	2.77	**2.6 to 2.8**
Water Absorption	0.17%	0.37%	**-**

3.2.3 Water

Water is essential element of concrete mix. The water for mixing and curing concrete should be free from unwanted harmful materials. Water to be used should be free from organic matter,silt, oil, sugar, chloride, and acidic material. Thus, potable water is usually used for this purpose. In the present study, potable tap water was used duringthe entire casting and curing period.

3.2.4 Superplasticizer

For proper workability, a water-reducing superplasticizer is required to be used. They are water-soluble polymers that dissolve in water to form long molecules that carry high negative charges. As the cement particles are dispersed by electrical repulsion between these negative charges, the combined water between the cement agglomerations gets freed and significantly improves the workability of concrete. Fosroc Auramix 200 conforming to (IS 9103 1999) and (ASTM C494 2005) was used as an admixture for the present study. The properties are listed below.

Table 3. 7 Properties of super plasticizer

Parameter	**Auramix 200**
Appearance	Yellowish to Brownish Liquid
pH	Minimum 6.0
Specific Gravity	1.03 – 1.07
Chloride Content	Nil (As per BS:5075 Part I)
Alkali Content	Typically, less than 1 g Na_2O equivalent/litre of admixture.

3.2.5 Carbon dioxide

The carbon dioxide gas used in this study was locally available, industrial, and 99.9 percent pure. Carbon dioxide (CO_2) is an odourless, colourless gas with a faintly acidic taste that is mildly poisonous. Industrial facilities that make hydrogen or ammonia from natural gas, coal, or other hydrocarbon feedstocks, as well as large-scale fermentation processes where plant products are converted into ethanol for use as a fuel for humans, automobiles, or industry, are the most frequent operations from which commercially produced carbon dioxide is recovered. Commercially, carbon dioxide is offered as high-pressure cylinder gas (about 300 psi or 20 bar).

3.3 Design mix proportions of concrete

The mix design was one of the most crucial aspects of this investigation. Because the injection of carbon dioxide tends to dry out the mix, the mix was designed with the w/c ratio in consideration. The M35 mix was designed with a w/c of 0.45 and prepared according to method given in (IS: 10262 2019) using the properties of materials as discussed above. Before finalising the mix design, many trials were conducted. As shown in Table 3.8, various trials with and without carbon dioxide and superplasticizer were conducted on the six cube samples of size 150 x 150 x 150 mm.

Table 3. 8 Concrete mix proportions for trails

Description	Mix 1	Mix 2	Mix 3	Mix 4
Cement Type	OPC	PPC	OPC	PPC
Cement (kg/m^3)	443	443	400	400
Water (kg/m^3)	195	195	182	182
Fine Aggregate (kg/m^3)	659	659	674.584	674.584
Coarse Aggregate (kg/m^3)	1131	1131	1200.63	1200.63
Super Plasticizer	None	None	0.65% by wt. of cement	0.65% by wt. of cement
Slump value (mm)	< 50	< 20	>150	>100
Cube Compressive Strength (7days)	20.2 MPa	16.6 MPa	23.5 MPa	19.7 MPa
Cube Compressive Strength (28days)	35.4 MPa	30.6 MPa	38.8 MPa	36.1 MPa

After the conclusion of the trails, based on the workability and strength parameters, the mix finalized for casting is shown in the Table 3.9.

Table 3. 9 Finalized mix design

Mix	Proportion
Cement	400 kg/m^3
Fine Aggregate	674.584 kg/m^3
Coarse Aggregate	1200.63 kg/m^3
Water	182 kg/m^3
Super Plasticizer	0.65% by weight of cement

After finalizing the mix design, the concrete was cast. A total of ten mix combinations with different binder types (OPC and PPC) and varying dosages of carbon dioxide (0%, 0.05%, 0.10%, 0.15%, 0.20%) were prepared by keeping the cement content, fine aggregate, coarse aggregate and water content constant in all mixes. The mix proportion of the ingredients is mentioned in Table 3.10.

Table 3. 10 Mix proportion of the ingredients (M35)

Cement	Fine Aggregate	Coarse Aggregate (10 mm)	Coarse Aggregate (20 mm)	Water
1	**1.68**	**1.20**	**1.80**	**0.45**

Table 3. 11 Concrete Mix Proportions

Mix Designation	Binder Type	Binder	Water	Coarse Aggregate	Fine Aggregate	Superplasticizer	CO_2 Content
		kg/m^3	kg/m^3	kg/m^3	kg/m^3	% By weight of cement	% By weight of cement
OPC CM	OPC	400	182	1200.63	674.584	0.65	0
OPC 0.05% CO_2	OPC	400	182	1200.63	674.584	0.65	0.05
OPC 0.10% CO_2	OPC	400	182	1200.63	674.584	0.65	0.10
OPC 0.15% CO_2	OPC	400	182	1200.63	674.584	0.65	0.15
OPC 0.20% CO_2	OPC	400	182	1200.63	674.584	0.65	0.20
PPC CM	PPC	400	182	1200.63	674.584	0.65	0
PPC 0.05% CO_2	PPC	400	182	1200.63	674.584	0.65	0.05
PPC 0.10% CO_2	PPC	400	182	1200.63	674.584	0.65	0.10
PPC 0.15% CO_2	PPC	400	182	1200.63	674.584	0.65	0.15
PPC 0.20% CO_2	PPC	400	182	1200.63	674.584	0.65	0.20

3.4 Mixing procedure and curing methodology

The ingredients of concrete were thoroughly mixed with the help rotary concrete mixer to achieve a uniform mix. In the beginning, drying mixing was done until a uniform blend was obtained. The water incorporated with the superplasticizer was added in phase wise manner. In mixes with carbon dioxide injection, after the complete addition of water into the mix, the mixer was sealed airtight with only a small opening for carbon dioxide injection see Figure 3.4 and Figure 3.5. The total mixing time was about 2-3 minutes. Before concrete placement in moulds, various tests were performed for each mix on freshly hydrating concrete. A vibrating table was used to compact the moulds. The specimens of different sizes were cast as per the testing methodology requirements, as shown in Table 3.13. All the specimens were kept in the moulds for 24 hours and then removed from moulds to be kept for curing. After demoulding and marking the samples, they were cured in water for 7, 28, and 56 days. The curing of samples was done in the curing tank. Curing should be done with extreme caution to ensure that the concrete specimens achieve the requisite strength. If the curing is not done properly, the concrete may lose moisture and hence be unable to acquire the appropriate strength.

Figure 3. 4 Concrete mixing setup

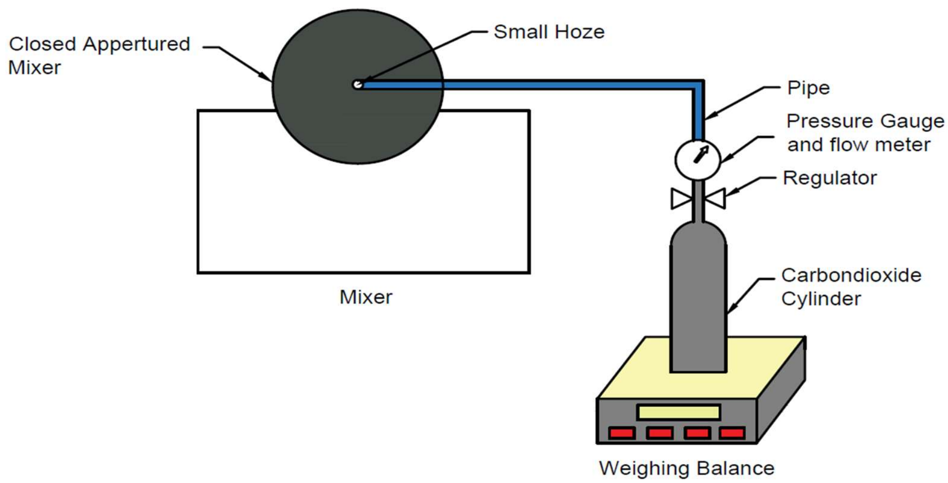


Figure 3. 5 concrete mixing setup block diagram

Two approaches were taken to measure the precise dosage of carbon dioxide: one by determining the flow rate vs. time relationship and two by weighing. Many trials were conducted to determine the flow rate vs. time relationship by filling the CO_2 into the balloons at different pressure rates. The time taken to fill the balloons by weight at different pressure rates was used to draw the relation between time and flow rate. To acquire more precession, the weighing balance was also utilized. The CO_2 cylinder was hefted on a weighing balance while injecting the carbon dioxide into concrete in the mixer, and the dosage quantity was accordingly injected. For this study, both approaches were taken to acquire the most precision. Flow rate vs. time relationship and weighing balance approach was simultaneously used. The carbon dioxide quantity to be injected was measured through both flow rate vs time relationship and weighing balance between acquiring the most precision.

After the curing period of 7, 28, and 56 days, the respective samples were removed from the curing tank for tests determining the mechanical properties, while for determining the durability properties, the curing period for the samples were set to 28 and 56 days.

Figure 3. 6 curing of samples in water tanks

3.5 Testing

The different tests performed on the casted specimens and standards followed for the testing procedure are as given below:

Table 3. 12 Tests performed and codes followed

Test	Code
Compressive strength test	**IS: 516 - 1959**
Split tensile strength test	**IS 5816: 1999**
Rapid chloride permeability test	**ASTM C1202 - 12**
Water absorption test	**ASTM C 642 - 97**
Sorptivity test	**ASTM C 1585 - 04**
Ultrasonic pulse velocity test	**IS 13311 (Part 1): 1992**
Rebound hammer test	**IS 13311 (Part 2): 1992**

Table 3. 13 Type and size of specimen for various tests performed

Name of the test	Type of specimen	Size of Specimen (mm)
Compressive-strength	Cube	**150 x 150 x 150**
Split-tensile-strength	Cylinder	**300 x 150 (L x D)**
Rebound Hammer	Cube	**150 x 150 x 150**
UPV	Cube	**150 x 150 x 150**
Sorptivity	Cylinder	**50 x 100 (L x D)**
Water Absorption	Cylinder	**50 x 100 (L x D)**
RCPT	Cylinder	**50 x 100 (L x D)**

The test setup for various fresh properties, mechanical properties, durability properties, and non-destructive properties of concrete are:

3.5.1 Fresh Properties

3.5.1.1 Workability of Concrete (Slump Test)

The slump test is one of the most accessible and popular tests used to determine the workability of the mix. The test was conducted as per (IS: 1199 (Part 2) 2018), as shown in Figure 3.7. For conducting the test,slump cone of an internal diameter of the slump cone at top is 10cm and at bottom, it is 20 cm with the height of the slump cone 30 cm. The mould is placed on horizontal and rigid surface. Mould was then filled in 3 layers with 25 tempering to each layer. Mould was slowly raised invertical direction and concrete is allowed to subside. The difference in levels between the height of the mould and that of the highest point of the subsided concrete was measured in mmand this was taken as slump value of the mix.

Figure 3. 7 Slump measurement of the concrete

3.5.1.2 Air Content Test

To study the void filling effect of carbon dioxide injection, air content in fresh concrete was determined by Water-Column method according to (IS: 1199 (Part 4) 2019). The apparatus is shown in Figure 3.8. The test is started within 15 minutes of obtaining the mix from the mixer. The test apparatus base is of cylindrical shape. The concrete is filled in three equal layers and each layer was tempered 25 times and the outside of the base is struck 10 to 15 times with mallet to close any air voids after each layer. The residual concrete is struck off and the top surface is levelled. The top of the apparatus is then latched on the base and the gap between struck off concrete and zero reading on the gauge is filled with water. The apparatus top is the pressurized with the built-in hand pump and the reading is recorded on the gauge (h1) and when the pressure is released another reading (h2) is recorded. If the reading h2 is 0.2 percent air content or less, then the value ($h1 - h2$) as the apparent air content (C_1). If $h2$ is greater than 0.2 percent air content, then the pressure is applied again, giving a new gauge reading $h3$ and a final reading $h4$ after the pressure is released. If ($h4 - h2$) is 0.1 percent air content or less, record the value ($h3 - h4$) as the apparent air content. After calculating the apparent air content, the aggregate correction factor (G) is subtracted from it to get the final value i.e. total air content.

$$Total\ Air\ Content = C_1 - G$$

Figure 3. 8 Air content test apparatus

3.5.1.3 Temperature

To ensure that fresh concrete meets acceptable temperature criteria, the temperature of the concrete was determined according to (ASTM C1064 2004). The temperature of the concrete is measured using a calibrated thermometer. The temperature of the concrete has an impact on how it cures and gains strength. As a result, it is vital to check the temperature of the concrete as it is being mixed and placed. After obtaining the mix from the mixer, the thermometer was placed into the mix in a way that its stem is completely inside the mix for the minimum of 2 minutes to record the reading.

Figure 3. 9 Temperature of the concrete

3.5.1.4 Density

The bulk density measurement is used for determining the weight per cubic meter of freshly mixed concrete (density of concrete) from which the yield of concrete per cubic meter can be calculated. Density for fresh concrete was calculated using the formula provided in (IS: 1199 (Part 3) 2018). To measure the density of fresh concrete, a cylindrical mould (as shown in Figure 3.10) of 25 cm diameter and 30 cm height was firstly weighed empty as W1. Then the concrete was filled in three equal layers with each layer tampered 25 times and top layer smoothed out. The filled cylinder was weighed as W2. Therefore, the total weight of the concrete W equals to (W2-W1). Then according to formula:

$$Density = \frac{Weight\ of\ the\ concrete\ in\ the\ cylinder}{Volume\ of\ the\ cylinder}$$

Figure 3. 10 Density of the concrete measurement

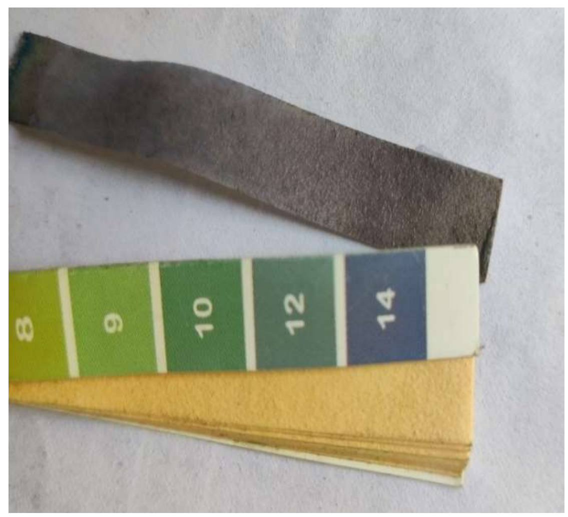
3.5.1.5 Setting Time

Setting of concrete is identified as the transition of fresh concrete from liquid phase to solid phase. It is important to identify this phase change to plan transporting and placing of concrete. Current practice of determining initial setting time of concrete is based on (ASTM C403 2008). According to this standard, initial setting time is identified based on the penetration resistance measured on mortar sieved from a concrete mixture and it is defined as the time taken to achieve a penetration resistance of 3.5 MPa. Fresh concrete mix is sieved using a 4.75 mm sieve to get a mortar sample. Three 150x150x150mm moulds were filled with sieved mortar mix. The moulds with mortar were then kept in an inclined position for 200 minutes, and bleed water collected on the surface of each specimen was removed with a pipet. Then, depending on the degree of setting of the mortar, a needle of sufficient size was placed into it. With the stiffening of mortar, a needle with the biggest diameter was employed at first, followed by needles with lesser sizes. The needles used have standard sizes of 645 mm, 323 mm, 161 mm, 65 mm, 32 mm, and 16 mm. A vertical force was exerted gradually and consistently downwards until the needle penetrated the mortar to a depth of 1 inch in 10s. The overall force was then recorded from the gauge. After that, the appropriate penetration resistance was estimated by dividing the overall force by the needle's contact area. The time of application force was measured as the elapsed time after the initial contact of cement and water. Finally, the graph of penetration resistance verses the elapsed time was plotted and the time corresponding to the penetration resistance value of 3.5 MPa and 27.6 were obtained. The time corresponding to these values are denoted as initial and final setting time of fresh concrete respectively.

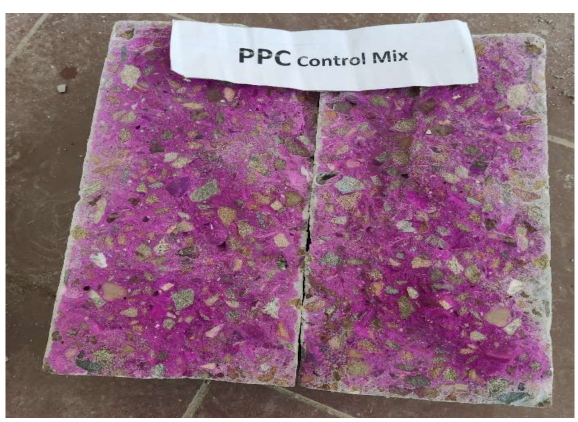
Figure 3. 11 Test apparatus for determining the setting time of the concrete

3.5.1.6 pH

The pH of concrete is an essential indicator of its alkalinity level. The most severe concrete damages are caused or accompanied by a drop in alkalinity and, as a result, a decrease in concrete's pH value. One of the most significant factors responsible for the reduction in pH value in the concrete is the carbonation. The carbon dioxide gradually penetrates the concrete and reacts with the hydration products, converting them into a mixture of calcite with lesser amounts of silica, alumina and iron oxides. In this study, the addition of carbon dioxide during mixing could be seen as the concern in terms of pH of concrete. Thus, it's critical to use an accurate and reliable method to determine the pH of concrete. The pH of concrete was determined by three different methods such as pH meter, pH paper, and Phenolphthalein Indicator. The pH meter and pH paper were utilized to determine the pH values of fresh concrete as well as of hardened concrete at different ages while the Phenolphthalein Indicator could only be utilized for hardened concrete at different ages.



(a) (b)



(c)

Figure 3. 12 Various methods for determining the pH of concrete (a) pH meter (b) pH paper (c) phenolphthalein indicator

3.5.2 Mechanical Properties

3.5.2.1 Compressive Strength

Compressive strength is the maximum compressive stress that a given solid material can sustain without fracture under a gradually applied load. This test was performed in accordance with (IS: 516 1959). The cubes are placed in such a way that the application of load is done to the opposite sides of the cubes as cast where the casting face and the testing face are perpendicular to each other. The load is axially applied without any shock and increased continuously at a loading rate 5 KN/s. The maximum load sustained by the specimen is recorded and divided by cross-sectional area to obtain the compressive strength of the specimen.

Concrete cubes of 150 x 150 x 150 mm dimension were casted to test for compressive strength and tested as specified in IS 516 (2004). The cubes were tested for optimum dosage at 7, 28 and 56 days of curing age. Figure 3.13 shows the testing of concrete specimen for compression.

For each type of concrete mix, three cubes were tested. For the compressive strength of the concretemix, the average value of three specimens was taken. The formula for calculating compressive strength is given as:

$$f_{ck} = \frac{P}{A}$$

where,

A = 150×150×150 mm size of cube's surface area (mm^2)

P = Maximum load applied (N)

f_{ck} = Compressive strength (N/mm^2)

Figure 3. 13 Testing of specimen for compression in CTM

3.5.2.2 Split tensile strength

Concrete is generally low in tensile strength and is brittle in nature, therefore is not usually expected to resist in direct tension. However, it is necessary to determine the tensile strength of concrete so as to identify the load at which the concrete members may crack. Cylindrical specimens of 150 mm diameter (D) and 300 mm height (l) were cast and tested according to (IS: 5816 1999). They are placed in the centering jig with a packing strip and/or loading pieces carefully positioned along the top and bottom of the plane of loading of the specimen. The load is to be applied without shock and increased continuously at a nominal rate within the range of 1.2 N/ (mm^2/min) to 2.4 N/ (mm^2/min). The maximum load (P) taken by the specimen before failure is noted, and the split tensile strength f_{ct} of the concrete cylinder is found from the formula in the equation below.

$$f_{ct} = \frac{2P}{\pi dl}$$

Where,

‘P’ is the maximum load in kN

‘l’ is the length of the cylinder in mm

‘d’ is the depth of the cylinder in mm

The cylinders were tested for optimum dosage at 7, 28, and 56 days of curing age. For each type of concrete mix, three cylinders were tested. For the split tensile strength of the concrete mix, the average value of three specimens was taken.

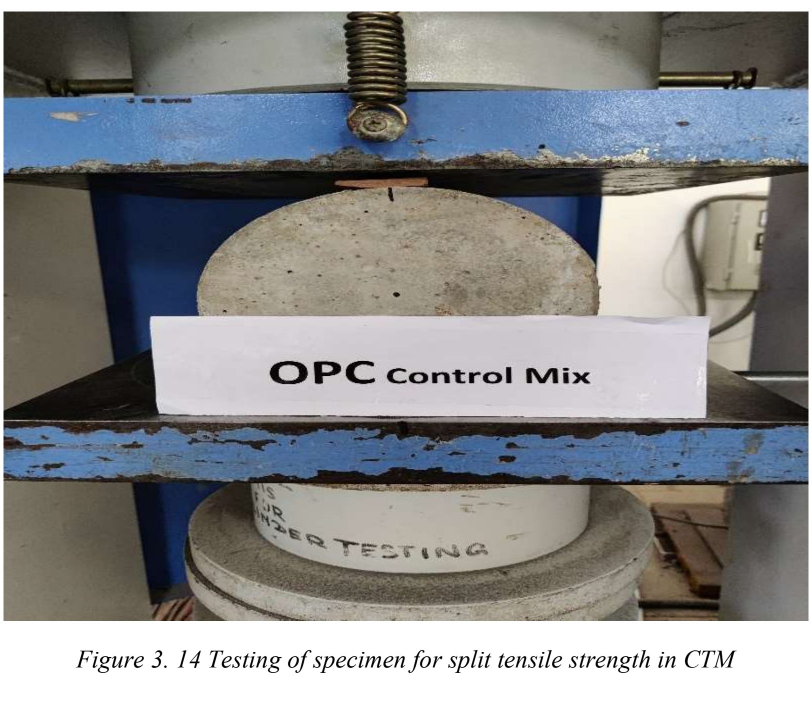


Figure 3. 14 Testing of specimen for split tensile strength in CTM

3.5.3 Durability Properties

The durability of concrete is a very important characteristic that should be considered in the evaluation. Durability mainly depends on the permeability of concrete. Water quality plays a very significant role in defining the durability of concrete.

3.5.3.1 Sorptivity

The performance of concrete exposed to extreme environments is a function of the degree of the penetrability of the pore system. Various variables determine the surface absorption in concrete such as mix proportions, admixture presence, voids, degree of hydration, type of placement, cementitious material chemical characteristics, etc.

The rate of absorption or sorptivity is measured using the procedures laid down in (ASTM C1585 2004). Here the mass differential of a specimen resulting from water absorption as a function of time was measured when only one surface of the specimen was exposed to water. Epoxy resin and polythene sheets were used to ensure one-side absorption. The method tests the susceptibility of unsaturated concrete to water penetration. Figure 4.20 shows the schematic diagram for testing the specimen. Samples were tested at the age of 28 and 56 days for water sorptivity. In this test method, cylindrical samples of height 50 mm and diameter of 100 mm were extracted by cutting the larger cylinder of 100 mm diameter and 200 mm height as shown in fig.3.10. These smaller samples were oven dried at a temperature of 50°C for three days and conditioned in airtight bags for 15 days. These smaller cylinders were coated with epoxy resin along the curved surface area. The top surface was covered with polythene sheet so that water was absorbed only by the uncovered bottom surface. Before placing the samples in contact with water, their initial mass was recorded. After taking the initial mass reading, samples were placed on wooden sticks to avoid direct contact of the sample surface with the tray. The tray was filled with tap water up to 3 mm above the top of the support device and this water level was maintained throughout the test period. Time was noted from the instant the samples were contacted with water. The change in mass of the samples was recorded at the time intervals mentioned in the code. The volume of water absorbed by the specimen per unit cross-section area of the specimen was calculated, and a plot was drawn between the square root of time and the volume of the water absorbed. The slope of the best-fit line gave the value of initial & secondary absorption to initial and secondary curves, respectively. While the constant of the best fit, the line equationgives the sorptivity coefficient. If the correlation coefficient (R) is less than 0.98 (the curve does not follow the linear relationship), then the water absorption rate cannot be determined.

For each type of concrete mix, three specimens were tested. For the sorptivity of the concrete mix, the average value of three specimens was taken. The formula for calculating the absorption is shown below:

$$I = \frac{mt}{a * d}$$

Where,

m_t = at time t, change in specimen mass (g)

I = the absorption (mm)

d = the density of water (g/mm^3)

a = the specimen's exposed area (mm^2)

(a)

(b)

(c)

Figure 3. 15 Sorptivity test setup (a) oven drying the samples (b) conditioning of samples (c) placing the epoxy coated samples in water

3.5.3.2 Rapid chloride permeability test

Rapid Chloride permeability test (RCPT) determines the concrete's electrical conductance, indicating its resistance to chloride ions penetration. The capacity of concrete to resist chloride ion penetration is measured using RCPT. The constant voltage (V) is supplied to a concrete specimen for 6 hours, and the current passing through the concrete is recorded to obtain the coulombs used for durability-based quality control. This test evaluates the electrical conductivity of various concrete mixes and reveals their resistance to chloride ion penetration. It measures the amount of electrical current that flows through concrete specimens during a set period. The movement of ions in a porous media under a concentration gradient is called diffusion. As a quality control measure and to assess advances in the qualities of new concrete, it is often crucial to determine the permeability of concrete to chloride ions.

This test was done as per the procedure shown in (ASTM C1202 2012). A concrete cylinder (diameter 100 mm and height 200 mm) was cut into slices of diameter 100 mm and height 50 mm to prepare samples for this test. This test is conducted through Rapid Chloride Permeability Test equipment. It consists of two reservoirs. One of them is filled with 3.0% of NaCl solution, and another reservoir is filled with 0.3N NaOH solution. This test was performed after the curing age of 28 and days, respectively. Firstly, the specimens were put into the vacuum desiccator's bowl. In the vacuum desiccator's bowl, the vacuum was maintained for 3 hours. The flow of de-aerated water was allowed into the desiccator to cover the concrete specimens and block the entry of air. After removing the concrete samples from the vacuum desiccator's bowl, they were surface dried and mounted into the gasket. Across the ends of the specimen, a potential difference of 60 Volt (DC) was provided. Two solutions were filled ineach reservoir of the gasket. Sodium chloride solution (3% NaCl solution) used for immersion of one end of the specimen, and Sodium hydroxide solution (0.3N NaOH solution) used for immersion of another end of the specimen. In this test, the amount of electrical current passed from the concrete specimen was monitored for 6 hours. And for measuring chloride ion penetration, "PROOVE it" data processor was used. According to Table, on the basis of the charge passed, the chloride ion penetrability was decided.

For each type of concrete mix, three specimens were tested. For the chloride ion penetrability of concrete mix, the average value of three specimens was taken.

Table 3. 14 Based on charge passed through the specimen, the extent of chloride ion penetrability of concrete as per ASTM C1202 - 12

Charge Passed (coulombs)	Chloride Ion Penetrability
> 4000	High
2000 – 4000	Moderate
1000 – 2000	Low
100 – 1000	Very Low
< 100	Negligible

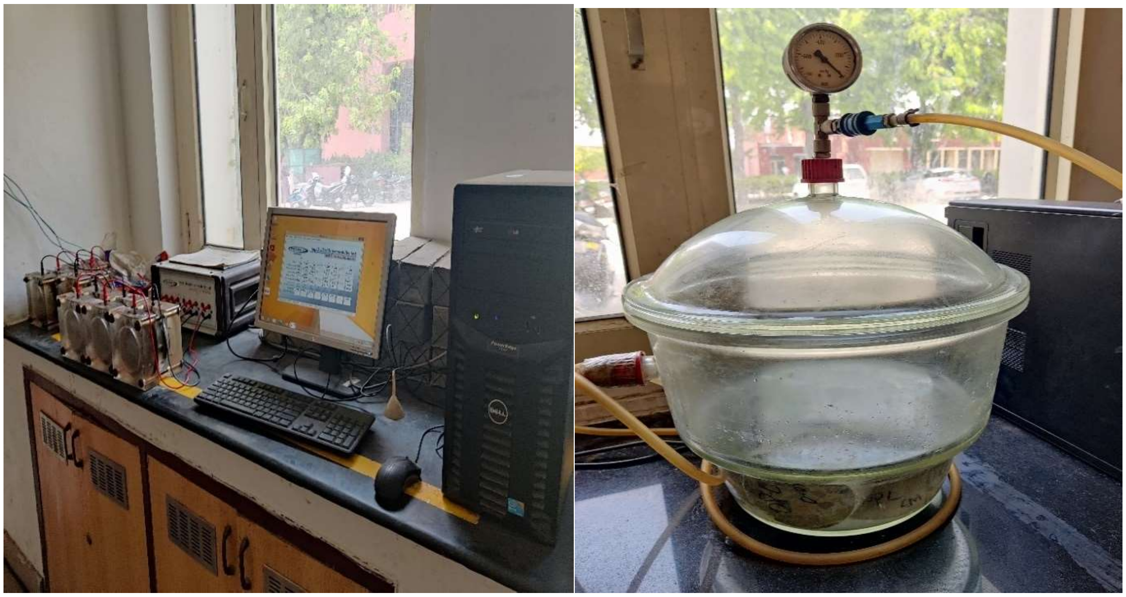


Figure 3. 16 Test setup for the rapid chloride permeability test with vacuum desiccator's bowl on right.

3.5.3.3 Water absorption test

Volume of permeable voids of concrete is an indirect measure of the porosity of concrete. It is the volumetric proportion of the voids in the concrete. The mechanical properties of the concrete are greatly influenced by its porosity and it is determined as per specifications of (ASTM C642 2013).

The concrete cubes, after curing were kept in the oven at a temperature of 110ºC for not less than 24 hours and weighed. This process was repeated until the difference between two successive readings was within 0.5% of the lesser weight. The specimens were then immersed in water at 21º C for not less than 48 hours and weighed after their surface was dried. The readings are taken till the difference between the two successive readings is less than 0.5%. After this step, the specimens are subjected to rigorous boiling in water for 5 hours and are allowed to cool under natural loss of heat for not less than 14 hours and then weighed. Finally, the specimens are suspended in water, and their apparent weights are taken. The volume of permeable voids (%) is determined by the formulae mentioned below in accordance with (ASTM C642 2013).

$$\text{Volume of permeable voids (\%)} = \left(\frac{(B-A)}{(B-C)}\right) \times 100$$

'A' is the mass of oven-dried sample in air, g;

'B' is the mass of surface-dry sample in the air after immersion and boiling, g;

'C' is the apparent mass of the sample in water after immersion and boiling, g

The samples were tested at the age of 28 and 56 days. For each type of concrete mix, three specimens were tested. For the volume of permeable voids of the concrete mix, the average value of three specimens was taken.

Figure 3. 17 Water absorption test: oven drying of samples (left) and immersion in boiling water (right)

3.5.4 Non-Destructive properties

3.5.4.1 Ultrasonic pulse velocity test

UPV is a popular non-destructive technique used to measure the homogeneity of the concrete. It is a convenient technique to assess the quality of concrete. If the concrete is more dense, homogenous, and uniform, relatively higher values of velocity are obtained. In case the quality of concrete is poor, lower velocities are obtained. The test was conducted on cubes after the curing age of 28 days using UPV of PROCEQ made with a 54 kHz transducer as per specification given in (IS: 13311-1 1992). The ultrasonic pulse velocity test device comprises a transmitter and a receiver positioned against two concrete sides. The equipment generates an ultrasonic frequency electronic pulse, which is transmitted through concrete via the transmitter. The transmitted pulses are picked up by the receiver, which is positioned on the other side of the concrete. When electronic pulses travel from transmitter to receiver, their travel time is measured. The average velocity of wave propagation (V) is calculated by dividing the length of the path travelled (L) by pulses by their travel duration (T). The pulse velocity is proportional to the strength of the concrete. The greater the pulse velocity, the greater the concrete strength.

$$V = \frac{L}{T}$$

For each type of concrete mix, three cubes were tested, and three readings were taken on each cube. An average of three readings was taken for each cube. The average value of three specimens was taken for the concrete mix pulse velocity. The apparatus used in this test is shown in Figure 3.18.

Table 3. 15 Concrete Quality Grading as per IS 13311 (Part 1): 1992

Pulse Velocity (m/s)	Concrete Quality Grading
Above 4500	Excellent
3500 – 4500	Good
3000 – 3500	Medium
Less than 3000	Doubtful

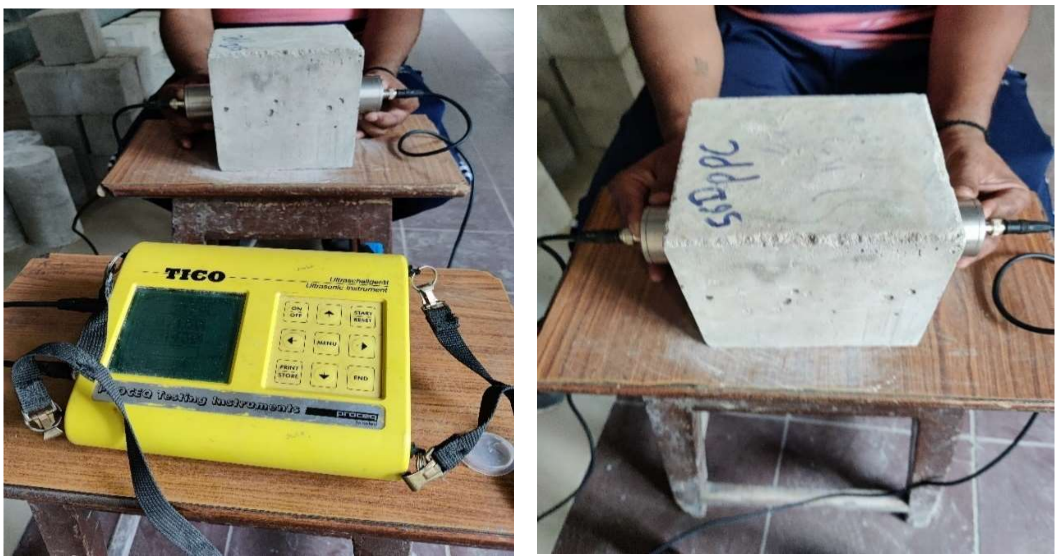


Figure 3. 18 Testing set up for determining ultrasonic pulse velocity through concrete

3.5.4.2 Rebound hammer test

The rebound hammer is used to measure the hardness of the surface of the concrete. It is a popular technique used to assess the strength of the concrete. A high rebound number indicates that the surface of the concrete is hard. Thus, it has higher strength, whereas a lower rebound number implies that the surface is relatively soft and concrete is weak. This test was conducted on cubes after the curing age of 28 days using a rebound hammer of PROCEQ made according to specifications in (IS: 13311-2 1992). The cube specimen was fixed from the top and bottom surface with the help of the jack, and the plunger was pressed against the concrete's surface (front and back). A gradual increase in pressure was applied until the hammer impacts.

For each type of concrete mix, three cubes were tested, and ten readings were taken on each cube (Five each from front and back surfaces); the point of impact should be 20 mm away, at least from any edge. An average of 10 readings was taken for each cube. And an average of three cubes was taken for the compressive strength value of the mix. Figure 3.19 shows the Rebound hammer used in the study.

Figure 3. 19 Performing the rebound hammer test on concrete specimen

3.5.5 Micro structural analysis

Scanning electron microscopic (SEM) analysis and X-Ray diffraction (XRD) were performed on powdered broken concrete samples for microscopic analysis of the effect of carbon dioxide addition on the properties of concrete samples.

3.5.5.1 Scanning electron microscopy (SEM)

Electron microscopy is one of the most powerful techniques for determining the microstructure of cementitious materials. To study the effect of carbon dioxide injection in concrete on cement chemistry, a detailed emphasis on the shape and microstructure is necessary. A high-energy beam of electrons is used to examine the objects in magnification in SEM. The formation of hydration products after hydration reaction is identified using the secondary electron mode of the SEM.

SEM was used to determine the elements in the carbonated concrete. In order to examine the micro structural change happening in the concrete during hydration when carbon dioxide is injected into the concrete, broken concrete samples were taken from the cubes tested for compressive strength. These samples were looked into in SEM for the possible densification and reduction in voids. Figure 3.20 shows the SEM equipment used in the present study.

Figure 3. 20 SEM equipment used in the study

3.5.5.2 X-Ray diffraction (XRD)

X-ray diffraction (XRD) is an important analytical tool for the characterization of cement. It is used for both qualitative and quantitative analysis. The basic concept on which it works is that the diffraction of X-rays by a crystalline material produces an XRD pattern consisting of peaks of varying intensities at characteristic diffraction angles. Patterns of peak positions and their relative intensities produced by XRD help characterize different crystal structures and identify their presence in unknown samples. The diffraction angle or position of the peaks is determined by the symmetry and the size of the unit cell through Bragg's law equation:

$$2d \sin \theta = n\lambda$$

Where,

'd' is the distance between lattice planes in the crystal in nm

'θ' is the incident/ diffracted angle in degree

'n' is an integer

'λ' is the wavelength of the scattered X-ray wave in nm

Powdered XRD analysis of the powdered samples was performed using Rigaku Minifelx II XRD equipment as shown in Figure 3.21 in the Physics Department of TIET Patiala with an X-ray source of Cu Kα radiation (λ = 1.5418 *Ȧ*) for diffraction angle varying from 5 to 90° in 2θ = 0.020°. The peak phases were identified using software named X'Pert HighScore Plus.

For micro structural analysis, the concrete powdered sample after sieving was placed on an acetone spatula and kept on the XRD machine. The scanning speed was fixed at 2^0 per minute, and 2θ ranged from 5-90^0.

Figure 3. 21 XRD equipment used in the study

CHAPTER 4

Results and Discussions

4.1 General

This chapter discusses results of various fresh properties, mechanical properties, durability properties, and non-destructive properties of all the concrete mixes with different dosages of carbon dioxide and binder types. All the tests conducted on the fresh, mechanical, durability, and non-destructive properties of these concrete mixes were in accordance with the test methods that were reported in Chapter 3.

4.2 Fresh Properties

An overview of the fresh properties is presented in Table 4.1. All the properties were deemed to be acceptable. There were slight changes in the fresh properties due to the addition of carbon dioxide, and the carbon dioxide-treated concrete samples were assessed to have performed equivalent to the control mix. The use of carbon dioxide had no negative effect on the properties of fresh concrete.

Table 4. 1 Fresh concrete properties

Mix Type	Temperature (°C)		Slump (mm)		pH		Unit Weight (kg/m3)		Air Content (%)	
CO_2 (%)	OPC	PPC	OPC	PPC	OPC	PPC	OPC	PPC	OPC	PPC
0	19	21.3	160	80	12.26	12.09	2389.47	2370.45	5.3	4.8
0.05	21	22.8	152	60	12.29	12.16	2354.14	2330.36	4.7	3.9
0.10	22.5	23.6	140	40	12.26	12.18	2341.91	2354.82	4.5	4
0.15	23.3	25.3	125	25	12.28	12.19	2369.09	2363.72	4.8	4.5
0.20	24.9	26.1	110	15	12.26	12.33	2386.07	2372.48	5.2	4.5

4.2.1 Slump

It was observed that with the increasing dosage of carbon dioxide, the slump of the mixture decreased due to the exothermic carbonation reaction. Thus, the concrete became drier with the percentage increase in carbon dioxide. The control mixes in both OPC and PPC had the highest slump compared to their respective carbon-treated mixes. As the mix was designed on the OPC binder, the mixes produced with OPC had a higher slump value and were more workable than the PPC-based mixes. Thus, an increase in the quantity of superplasticizers is required to achieve a higher slump value and increase the workability of PPC-based mixes. Based on the observations, the OPC mix with 0.10% CO_2 was soft and easily workable. The slump comparison of OPC and PPC-based mixes are shown in Figure 4.1.

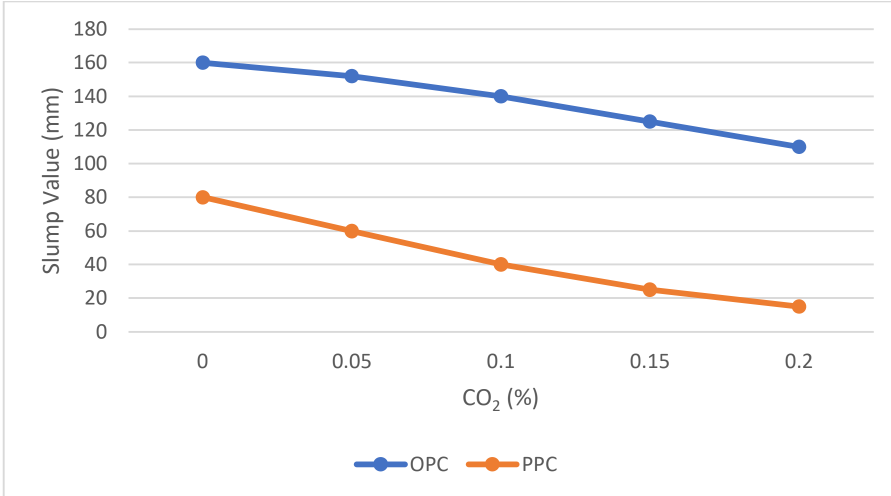


Figure 4. 1 Slump values vs CO_2 percentage

4.2.2 Temperature

It was observed that with the increasing dosage of carbon dioxide, the mix temperature increased. The temperature increase is expected due to the exothermic nature of the carbonation reaction; it is unclear whether the observed effects were entirely due to the carbonation process or due to other factors such as time between measurements, mixing, hydration process, increasing ambient temperature, etc.

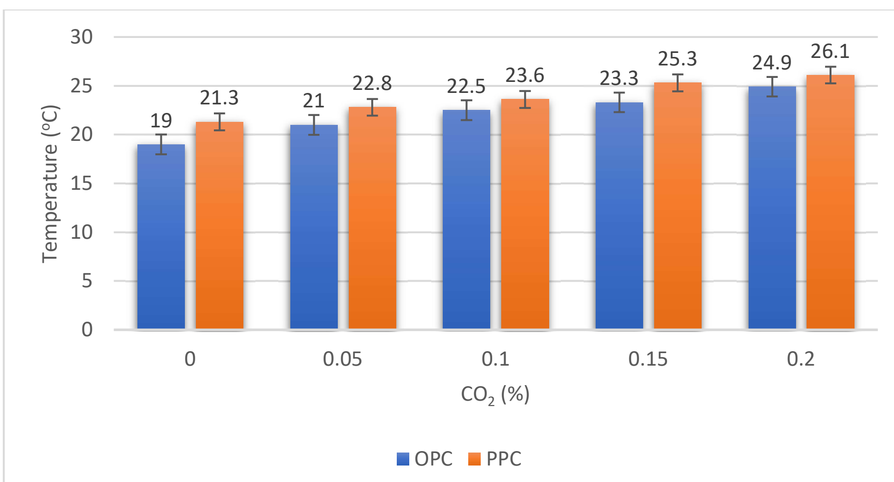


Figure 4. 2 Temperature vs CO_2 percentage

4.2.3 Unit Weight

No significant effect of the addition of carbon dioxide on density values was observed for both PPC and OPC-based mixes. The density tends to increase first and then decrease, as shown in Figure 4.3. This is most likely due to nanosized $CaCO_3$ formed during the reaction, which fills the pores of the concrete samples. For OPC-based mixes, the minimum density was observed for the control mix, while the 0.10% CO_2 mix had the highest, a 2% increase. And for PPC-based mixes, the minimum density was seen in the control mix, while the 0.05% CO_2 mix had the highest, a 1.7% increase.

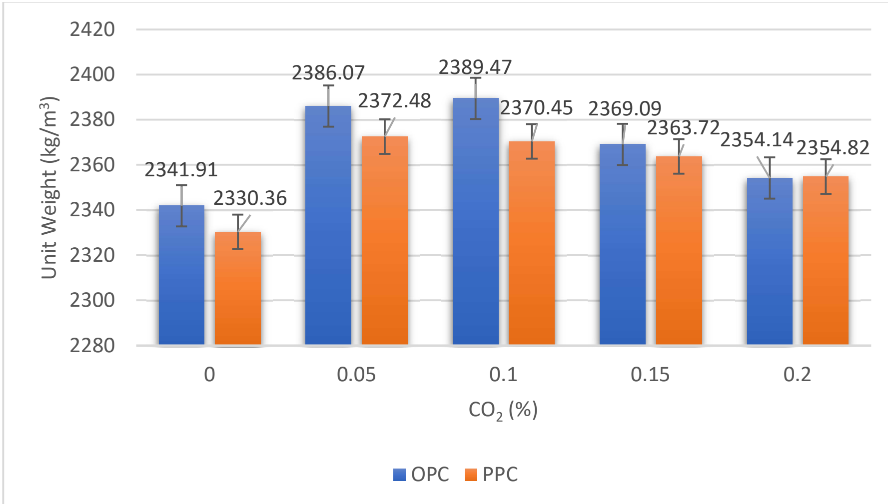


Figure 4. 3 Unit weight vs CO_2 percentage

4.2.4 Air Content

Percentage air content in fresh concrete was found to be highest in the control mix for both OPC and PPC-based mixes and the lowest at 0.10% CO_2 for OPC-based mixes and 0.05% CO_2 for PPC-based mixes. Three trials for each percentage were conducted, and an average was recorded. The trend is shown in Figure 4.4. The reduction in percentage air content was significant, thus showing that adding carbon dioxide during mixing helps provide a denser and more compact mix, which was also evident from the Figure 4.3. Also, the percentage of air content was less in PPC-based mixes than that of OPC-based mixes due to the varying particle size and presence of finer particles in PPC.

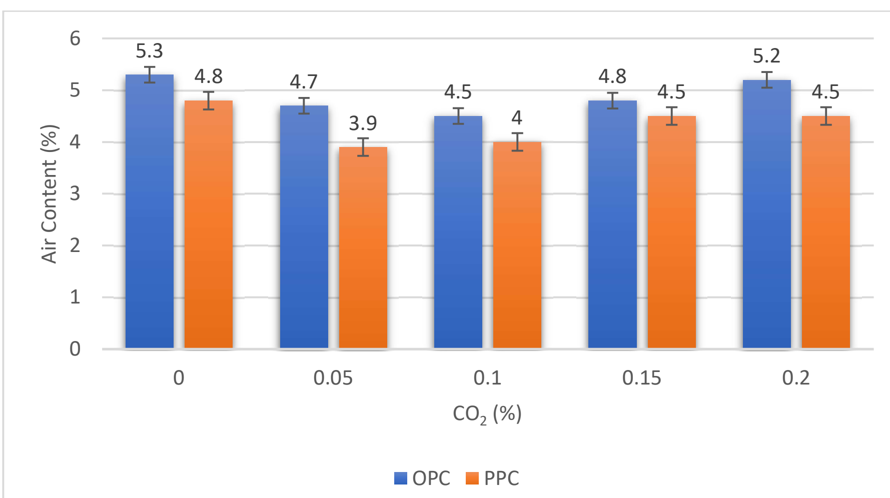


Figure 4. 4 Air content percentage vs CO_2 percentage

4.2.5 pH

The carbon dioxide in concrete is said to be a durability issue. One of the most significant factors responsible for reducing pH value in the concrete is carbon dioxide, like atmospheric carbonation. However, adding carbon dioxide to the fresh concrete might raise some eyebrows (durability concerns). Thus, pH has become one of the most important factors to be investigated, especially for carbon-treated mixes. The fresh concrete's pH was measured by the pH paper method and pH meter.

The pH paper turned greyish when dipped into the filtered fresh concrete and distilled water mixture. A similar greyish colour was seen for all carbonated or non-carbonated mixes. Thus, indicating the pH value of 12 to 14, i.e., basic. While from the pH meter, accurate values were recorded, as shown in Figure 4.5.

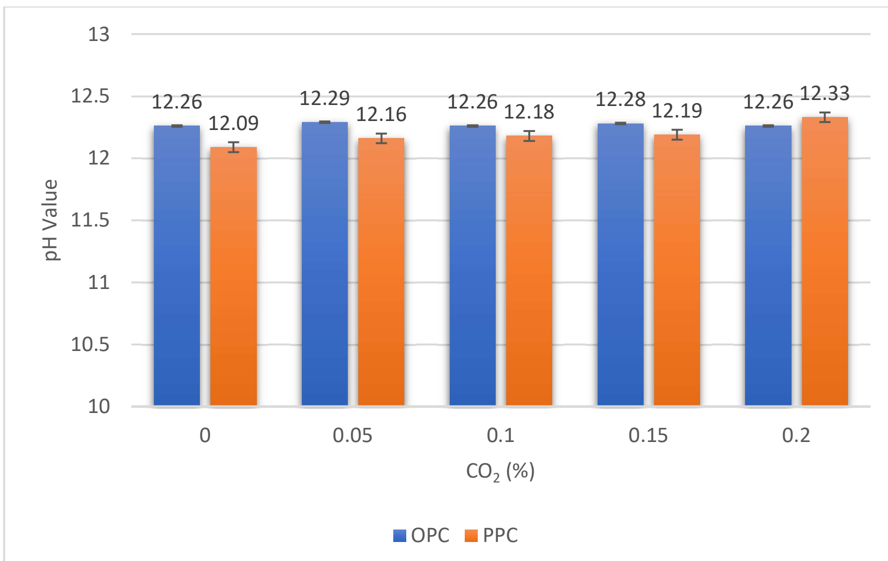


Figure 4. 5 pH values vs CO_2 percentage

There was no major change in the pH values with the addition of carbon dioxide. The pH of the concrete was within the permissible limits of 12 to 13.

4.2.6 Setting Time

Table 4. 2 Times of set

OPC							
Mix Type	**Initial set**				**Final set**		
CO_2 (%)	*Time (h)*	*Difference (min)*	*Relative to reference*		*Time (h)*	*Difference (min)*	*Relative to reference*
0	6:55		100.00%		9:38		100.00%
0.05	6:05	-50	87.95%		8:42	-56	90.31%
0.10	5:40	-75	81.92%		7:55	-103	82.17%
0.15	6:10	-45	89.15%		8:28	-70	87.88%
0.20	6:40	-15	96.38%		9:05	-33	94.29%
PPC							
Mix Type	**Initial set**				**Final set**		
CO_2 (%)	*Time (h)*	*Difference (min)*	*Relative to reference*		*Time (h)*	*Difference (min)*	*Relative to reference*
0	5:05		100.00%		7:42		100.00%
0.05	3:40	-85	72.13%		5:35	-127	72.51%
0.10	4:08	-57	81.31%		5:58	-104	77.48%
0.15	4:26	-39	87.21%		6:29	-73	84.19%
0.20	4:54	-11	96.39%		7:07	-35	92.42%

The results of the setting time are presented in Table 4.2. For each condition, the initial and final setting times are presented along with comparisons to the reference in terms of the actual differences (in minutes) and relative comparison. For the OPC-based mixes, the control mix had the highest setting time of 415 minutes (initial) and 578 minutes (final). The addition of CO_2 dosages reduced the time of the initial set between 15 and 75 minutes (4-18% reduction) and the final set by 33-103 minutes (6-18%) reduction. Amongst the carbonated mixes, the mix with 0.10% CO_2 had the least setting time of 340 minutes (initial) and 475 minutes (final) as shown in Figure 4.6. And for the PPC-based mixes, the control mix had the highest setting time of 305 minutes (initial) and 462 minutes (final). The addition of carbon dioxide accelerated the setting and reduced the time of the initial set by 11 to 85 minutes (4-18% reduction) and the final set between 35-127 minutes (8-18% reduction). The CO_2 dosage of 0.05% had the least initial set of 220 minutes and final set of 335 minutes among the other carbonated PPC-based mixes, as shown in Figure 4.7.

The observed acceleration in setting time with all CO_2 dosages might be due to the formation of nanoscale carbonation reaction products (Calcium Carbonates) that act as heterogeneous nucleation sites for hydration product precipitation from pore solution. Amongst the OPC and PPC-based mixes, the PPC-based mixes tend to set quicker than the OPC base mixes because of two main reasons. One is due to the varying particle size and presence of finer particles than

OPC. Two, the carbonation reaction and the presence of finer particles accelerates the hydration reaction, resulting in a high temperature (as shown in Figure 4.2) and a quick setting.

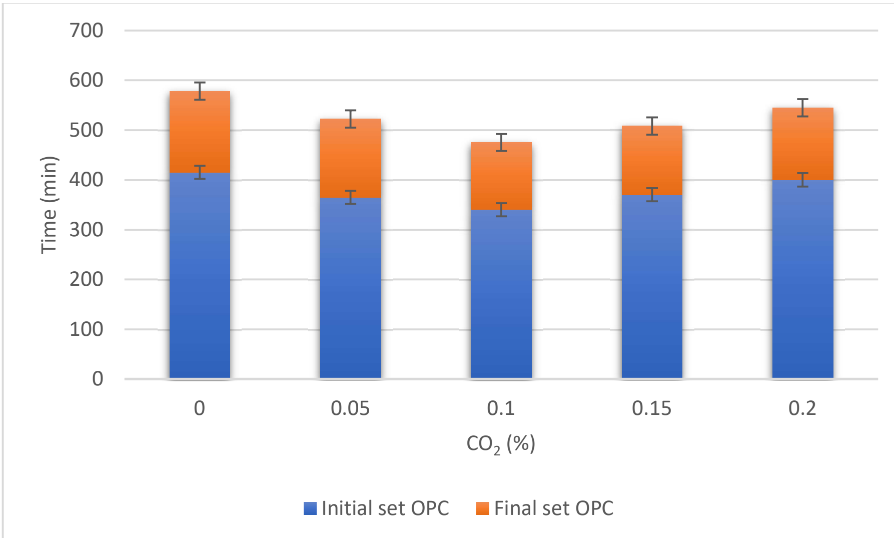


Figure 4. 6 Setting time vs CO_2 percentage (for OPC based mixes)

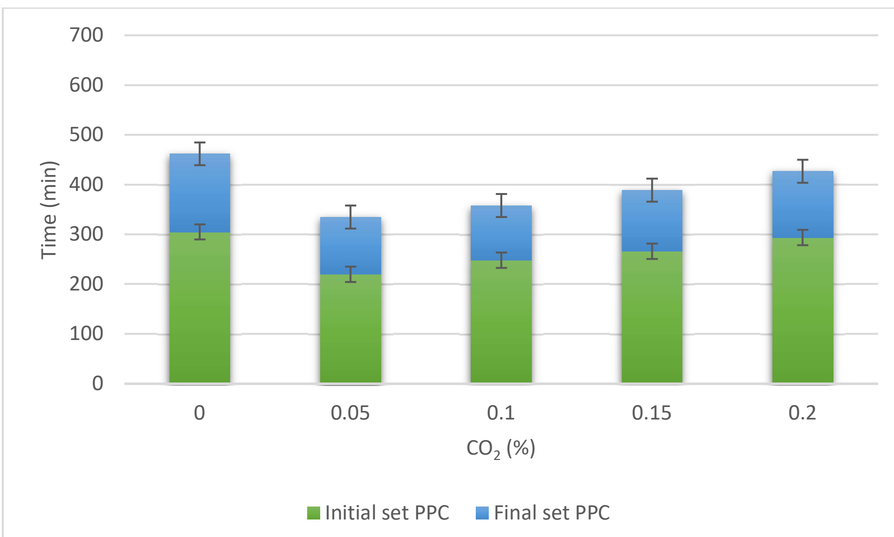


Figure 4. 7 Setting time vs CO_2 percentage (for PPC based mixes)

4.3 Hardened properties

The hardened properties of concrete are found on concrete after its final setting and after its curing for 7 days, 28days, and 56days. The different hardened properties tests are compressive strength of concrete, Split-tensile strength of concrete.

4.3.1 Compressive Strength

Table 4.3 depicts the compressive strength test results at different curing ages and different dosages of carbon dioxide with comparisons to the reference in terms of the actual differences and relative comparison. The average strength values for each condition are the average of three specimens.

Table 4. 3 Compressive strength results

OPC									
Mix Type	**7 Days**			**28 Days**			**56 Days**		
CO_2 (%)	*Compressive Strength (MPa)*	*Difference (MPa)*	*Relative to Reference*	*Compressive Strength (MPa)*	*Difference (MPa)*	*Relative to Reference*	*Compressive Strength (MPa)*	*Difference (MPa)*	*Relative to Reference*
0	24.25		100.00%	37.625		100.00%	39.125		100.00%
0.05	26.833	2.5833	110.65%	39	1.375	103.65%	44.937	5.812	114.85%
0.10	30.666	6.4166	126.46%	41.25	3.625	109.63%	50.583	11.458	129.28%
0.15	26.5	2.25	109.27%	36.875	-0.75	98.01%	42.158	3.033	107.75%
0.20	24.8	0.55	102.26%	35	-2.625	93.02%	36.625	-2.5	93.61%
PPC									
Mix Type	**7 Days**			**28 Days**			**56 Days**		
CO_2 (%)	*Compressive Strength (MPa)*	*Difference (MPa)*	*Relative to Reference*	*Compressive Strength (MPa)*	*Difference (MPa)*	*Relative to Reference*	*Compressive Strength (MPa)*	*Difference (MPa)*	*Relative to Reference*
0	23.8		100.00%	41.8333		100.00%	45.375		100.00%
0.05	28.58	4.783	120.09%	49.923	8.089	119.33%	55.875	10.5	123.14%
0.10	24.25	0.45	101.89%	44.2083	2.375	105.67%	49.125	3.75	108.26%
0.15	21.59	-2.21	90.71%	39.519	-2.3143	94.46%	46.1	0.725	101.59%
0.20	20.8	-3	87.39%	35.0416	-6.7917	83.76%	42.6	-2.775	93.88%

It has been observed that the addition of carbon dioxide helps to increase the compressive strength of the concrete. The compressive strength benefits of up to 25% can be achieved with the addition of carbon dioxide. The best compressive strength results for the OPC-based mixes came from the 0.10% CO_2, which provided a 26% improvement in strength at 7 days. The strength improvement of 10% and 29% were also achieved at 28 days and 56 days, respectively. The best compressive strength was recorded at 0.05% CO_2 for the PPC-based mixes. The strength improvement by 20% at 7 days, 19% at 28 days, and 23% at 56 days was achieved by the addition of carbon dioxide dosage. This improvement in the strength is due to the reaction between hydrating cement and the carbon dioxide, which results in the formation of nano-scale calcium carbonates that densifies the concrete by filling up the voids and improving the strength.

Figure 4.8 and Figure 4.9 shows the compressive strength comparisons of various mixes with different dosages of carbon dioxide and different binder types. It is evident from Figure 4.8 and

Figure 4.9 that the concrete strength decreased slightly with increasing CO_2 dose. The results suggested that for all curing periods, there is a gradual increase in compressive strength at 0.10% CO_2 for OPC-based mixes and 0.05% CO_2 for PPC-based mixes. A decrease in strength is observed after 0.05% CO_2 for PPC-based mixes and 0.10% CO_2 for OPC-based mixes. An adequate dosage of carbon dioxide may offer a well-balanced addition of nuclei to the system, whereas an excessive quantity may prevent future hydration. Initially, the reaction might occur in the pore solution; however, when additional carbon dioxide is added, the number of $CO3^{2-}$ ions in the solution increases, and the Ca^{2+} may not be replaced at the same pace. The carbon dioxide that reacts later may preferentially interact with Ca^{2+} at or near active dissolving sites rather than at a distance and in solution. The formation of reaction products, such as carbonates and any material that forms around such nuclei, may impede the affected site's ability to participate in hydration in the anhydrous phase.

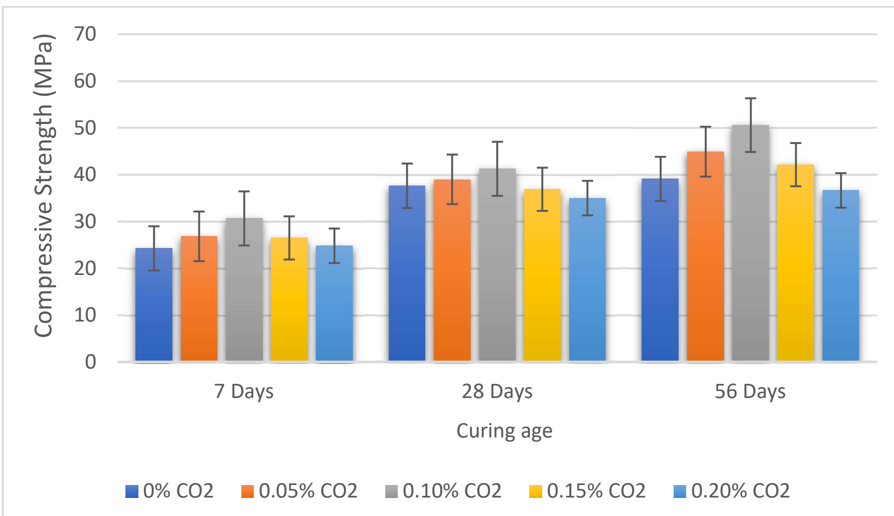


Figure 4. 8 Compressive strengths at 7, 28, and 56 days (for OPC based mixes)

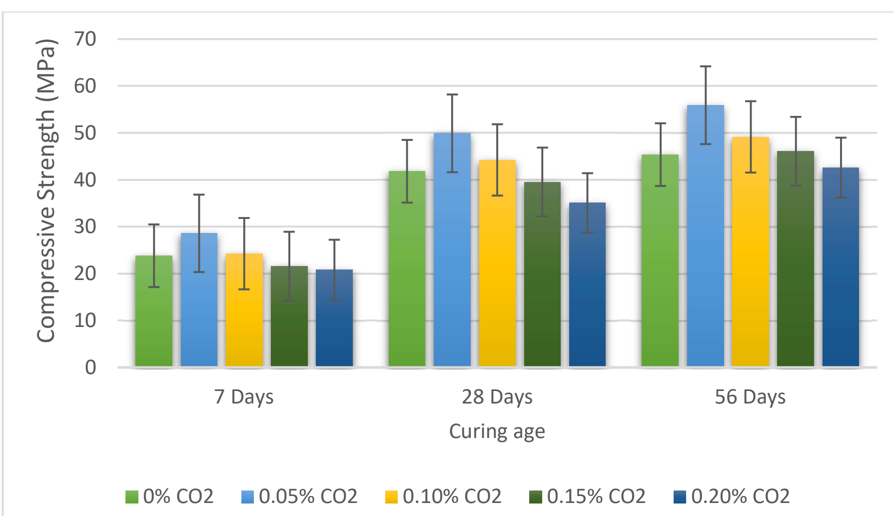


Figure 4. 9 Compressive strengths at 7, 28, and 56 days (for PPC based mixes)

4.3.2 Split Tensile Strength

The average Split-tensile strength of the concrete at different curing ages and different dosages of carbon dioxide with comparisons to the reference in terms of the actual differences and relative comparison is represented in Table 4.4:

Table 4. 4 Split tensile strength results

OPC									
Mix Type	**7 Days**			**28 Days**			**56 Days**		
CO_2 (%)	*Split Tensile strength (MPa)*	*Difference (MPa)*	*Relative to Reference*	*Split Tensile strength (MPa)*	*Difference (MPa)*	*Relative to Reference*	*Split Tensile strength (MPa)*	*Difference (MPa)*	*Relative to Reference*
0	2.862		100.00%	3.0281		100.00%	3.7028		100.00%
0.05	3.218	0.355	112.42%	3.305	0.2769	109.14%	4.0425	0.3397	109.17%
0.10	3.355	0.493	117.22%	3.9316	0.9035	129.83%	4.1883	0.4855	113.11%
0.15	2.791	-0.071	97.49%	3.4173	0.3892	112.85%	4.0631	0.3603	109.73%
0.20	2.548	-0.314	89.00%	3.0481	0.02	100.66%	3.9278	0.225	106.07%
PPC									
Mix Type	**7 Days**			**28 Days**			**56 Days**		
CO_2 (%)	*Split Tensile strength (MPa)*	*Difference (MPa)*	*Relative to Reference*	*Split Tensile strength (MPa)*	*Difference (MPa)*	*Relative to Reference*	*Split Tensile strength (MPa)*	*Difference (MPa)*	*Relative to Reference*
0	2.315		100.00%	2.9729		100.00%	3.448		100.00%
0.05	2.465	0.149	106.46%	3.8217	0.8488	128.55%	4.1146	0.6666	119.33%
0.10	1.992	-0.322	86.05%	3.6745	0.7016	123.59%	3.9717	0.5237	115.18%
0.15	1.596	-0.719	68.94%	3.448	0.4751	115.98%	3.5858	0.1378	103.99%
0.20	1.548	-0.767	66.87%	3.373	0.4001	113.45%	3.5193	0.0713	102.06%

It is evident from the results of the split tensile strength test of concrete that with an increase in dosage of carbon dioxide, the strength increases firstly and then tends to fall. The increase in strength by 13-30% at all curing ages is observed at 0.10% CO_2 in OPC-based mixes and 6-28% at 0.05% CO_2 at all curing ages for PPC-based mixes. This indicates low porosity and better strength between the cement matrix and interfacial transition zone. The results obtained are somewhat similar to that for compressive strength. Maximum tensile strength achieved is at 0.10% CO_2 in OPC-based mixes and 0.05% CO_2 for PPC-based mixes, beyond which the strength starts reducing.

Tensile strength is one of the key properties of concrete. Concrete is weak in tension. The brittle nature of concrete demands evaluation of tensile strength to determine the load of cracking. The variation in split tensile strength with different dosages of carbon dioxide, at various curing ages, and with varying types of the binder are shown in Figure 4.10 and Figure 4.11. Figure 4.10 shows the variations at different curing ages compared to the control mix for OPC-based mixes, while Figure 4.11 shows the variations with reference to the control mix for PPC-based mixes.

The study finds a gradual increase in split tensile strength for all curing periods at 0.10% CO_2 for OPC-based mixes and 0.05% CO_2 for PPC-based mixes. A decrease in strength is observed after 0.05% CO_2 for PPC-based mixes and 0.10% CO_2 for OPC-based mixes. At 7-day curing, the split tensile strength at 0.10% CO_2 is approximately 17% higher than a control mix for OPC-based mix, approximately 30% higher than a control mix at 28 days of curing, and approximately 13% higher than a control mix. While for PPC-based mix, approximately 6.5% increase in split tensile strength than a control mix is achieved for 0.05% CO_2 at 7 days curing, 28.5% increase than a control mix at 28 days curing, and approximately 19.5% increase than a control mix at 56 days curing. The split tensile strength beyond 0.05% CO_2 for PPC-based mixes and 0.10% CO_2 for OPC-based mixes tend to reduce. The loss in strength at 7-day curing is up to 11% and 33% compared to the control mix for OPC and PPC-based mixes, respectively. The split tensile strength beyond 0.05% CO_2 for PPC-based mixes and 0.10% CO_2 for OPC-based mixes at the curing ages of 28 and 56 days tend to fall compared to the peak values but is more than that of the control mix. The carbonation densifies the concrete mix and improves the interfacial transition zone (ITZ), which increases its split tensile strength.

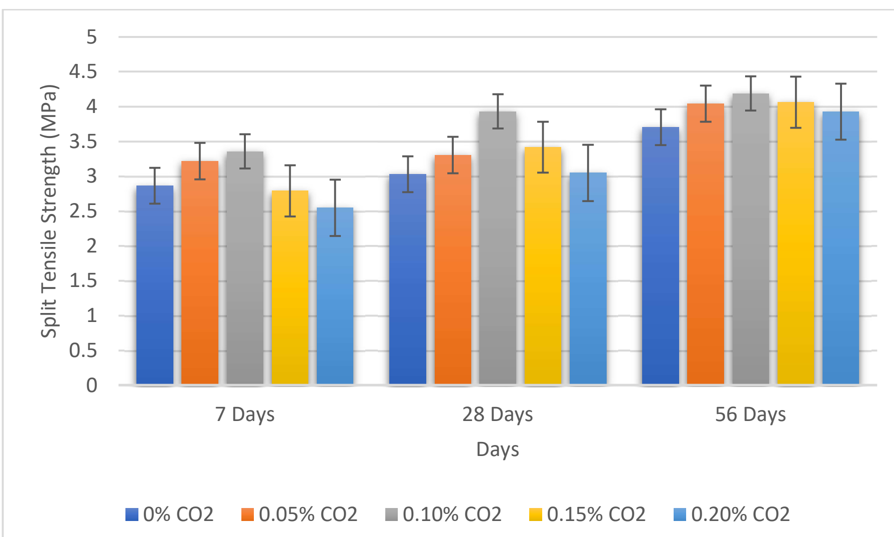


Figure 4. 10 Split tensile strengths at 7, 28, and 56 days (for OPC based mixes)

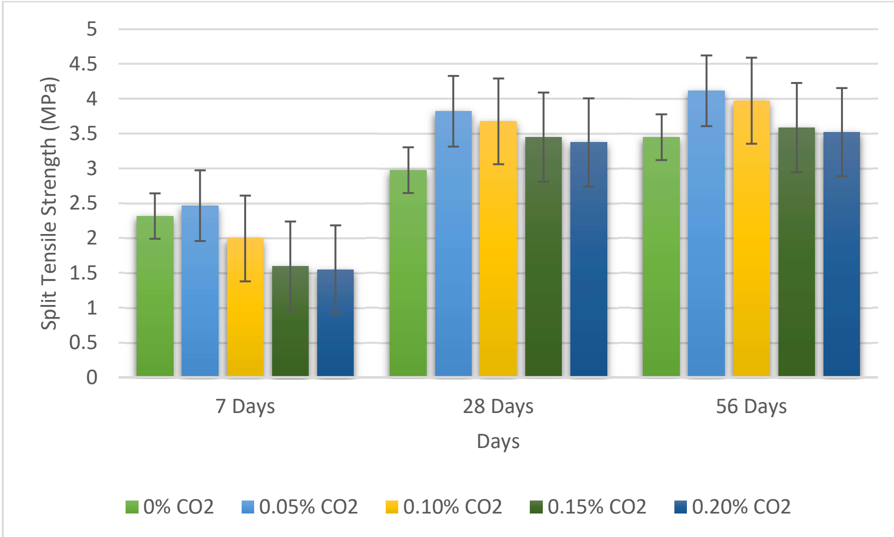


Figure 4. 11 Split tensile strengths at 7, 28, and 56 days (for PPC based mixes)

4.4 Durability

The addition of carbon dioxide in the concrete is said to be a durability concern. Thus, to rectify these concerns, various durability tests were conducted to check the effect of adding carbon dioxide to the concrete. Various tests conducted to evaluate durability were Rapid Chloride Permeability Test, Sorptivity Test, and Water Absorption Test. The observation of these tests is discussed in this section.

4.4.1 Rapid Chloride Permeability Test (RCPT)

The rapid chloride permeability test (RCPT) is a popular method for determining the durability of concrete, especially its resistance to chloride ion penetrability. The RCPT was performed in accordance with ASTM C1202. The test specimens were concrete discs with a diameter of 100 mm and a thickness of 50 mm made from cylindrical concrete specimens with a diameter of 100 mm and a height of 200 mm. The specimens were stored in the wet chamber until the test day. The 50 mm thick discs were made by cutting the 200 mm tall cylinder at the completion of the requisite curing days.

The test was conducted on 28 and 56 days of curing for all the concrete mixes. Each test result is the average of three specimens. The RCPT test results are presented in Table 4.5.

Table 4. 5 Charge passed (coulombs) in the Rapid Chloride Permeability Test (RCPT)

Mix Type	**28 Days**		**56 Days**	
CO_2 (%)	OPC	PPC	OPC	PPC
0	2849.333	1742	2471.333	1060
0.05	2572	1539.5	2531.667	827
0.10	2242.333	2056.667	2011.667	986
0.15	2467.667	2459.667	2311	1071.667
0.20	2683	2936	2627	1259.667

It is clear from Table 4.5 that with the addition of precise carbon dioxide dosage, the charge passed decreases. Correspondingly, there is an improvement in the concrete quality. Further, as the curing duration increases, there is an improvement in the concrete quality. The concrete with 0.10% CO_2 exhibited the best results for OPC-based mixes, while the best results were exhibited at 0.05% CO_2 for PPC-based mixes.

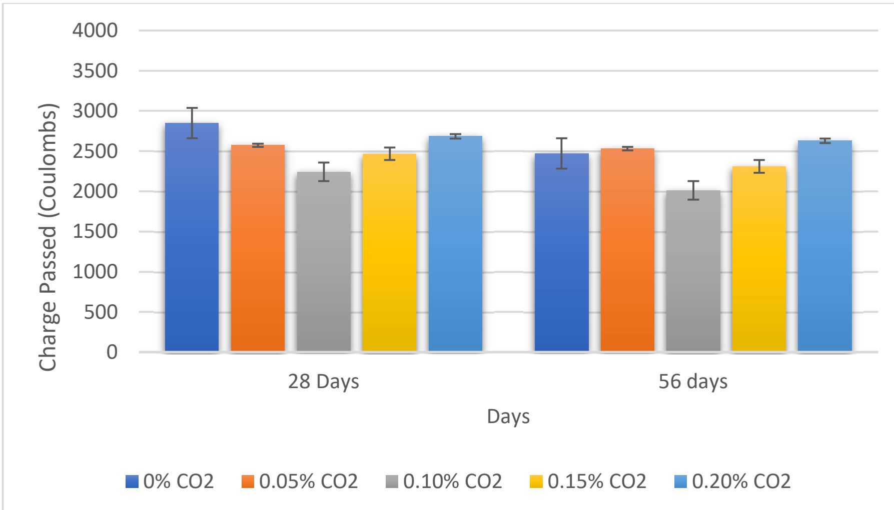


Figure 4. 12 Charge passed at 28 and 56 days (for OPC based mixes)

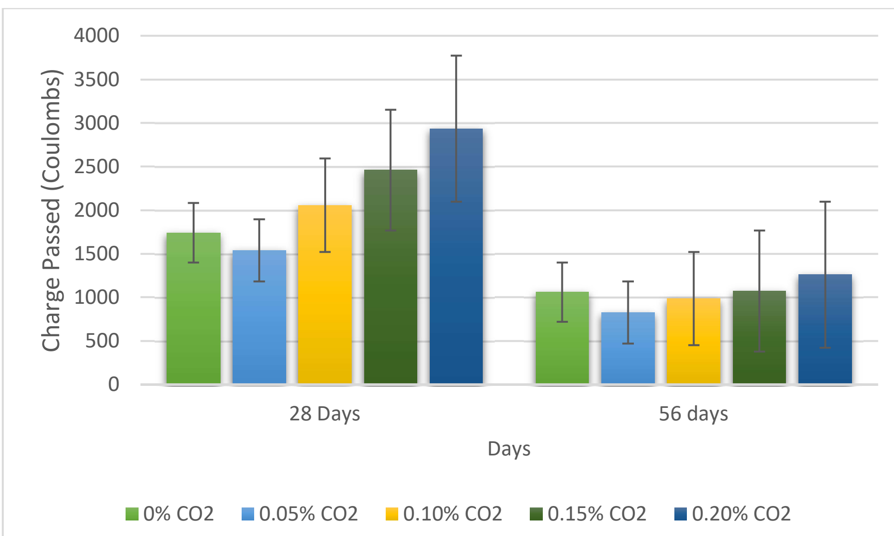


Figure 4. 13 Charge passed at 28 and 56 days (for PPC based mixes)

It is evident from Figure 4.12 and Figure 4.13 that with the addition of carbon dioxide, the charge passing firstly reduces and then increases with the increase in the dosage of carbon dioxide. The reduction in charge passing is due to the carbonation reaction, which tends to lower the porosity by densifying the concrete mix and improving the interfacial transition zone (ITZ). For the OPC-based mixes, the moderate charge passes through the concrete, and low to moderate charge is passed through the PPC-based concrete mix. The charge passing was least in the concrete with 0.10% CO_2 for OPC-based mixes. A 19-22% reduction in charge passing was recorded. The least charge passed through 0.05% CO_2 concrete for the PPC-based mixes. The decrease in the charge passing was about 12-22%. Also, it can be seen that the charge passing through the PPC-based mixes is much lower than that of the OPC-based mixes. It is due to the varying particle size and presence of finer particles than OPC that fill up the voids and reduces their porosity.

4.4.2 Water Absorption

The pore structure of concrete is quite well known to be critical to the material's durability. A simple test for characterization of this pore structure is widely investigated to develop a very simple compliance requirement for concrete durability. Water absorption by immersion is regarded as an essential criterion in this regard. The table presents the water absorption and volume of voids for different concrete mixes.

Table 4. 6 Water absorption test results

28 Days						
Mix Type	**Absorption after immersion**		**Absorption after immersion and boiling**		**Volume of permeable pore space (voids), %**	
CO_2 (%)	OPC	PPC	OPC	PPC	OPC	PPC
0	4.709	5.275	4.460	5.203	10.416	11.734
0.05	4.974	5.055	4.939	5.065	10.630	11.095
0.1	4.244	6.027	4.096	5.904	9.594	12.763
0.15	4.828	6.140	4.808	6.193	10.319	13.122
0.2	5.492	6.268	5.398	6.422	12.140	13.573
56 Days						
Mix Type	**Absorption after immersion**		**Absorption after immersion and boiling**		**Volume of permeable pore space (voids), %**	
CO_2 (%)	OPC	PPC	OPC	PPC	OPC	PPC
0	4.605	5.154	4.747	5.181	10.915	11.604
0.05	4.643	4.952	4.773	4.980	10.954	11.203
0.1	4.222	5.080	4.362	5.072	10.085	11.447
0.15	4.453	5.358	4.531	5.408	10.316	12.043
0.2	5.277	5.878	5.358	5.905	12.066	13.109

It has been observed that water absorption decreases with the addition of precise dosage of carbon dioxide. For the OPC-based mixes, with the addition of 0.10% CO_2, the absorption after immersion was reduced up to 10%, absorption after immersion and boiling was reduced by 9%, and the volume of voids was reduced by 8% across all curing ages. For the PPC-based mixes, the mix with 0.05% CO_2 had the least water absorption. For 0.05% CO_2, the absorption after immersion was reduced by 5%, absorption after immersion and boiling was reduced by 4%, and the volume of voids was reduced up to 6% across all curing ages. The addition of carbon dioxide in the concrete results in the formation of nano-sized calcium carbonates, which impacts the pores of the concrete matrix by blocking the voids and gaps. The pore continuum and volume reduction result in reduced absorption of these concrete mixes.

Figure 4.14 to Figure 4.19 shows the water absorption percentage and percentage volume of voids of the different concrete mixes containing different CO_2 dosage and binder types at 28 days and 56 days of curing. All the figures show a similar trend. It can be seen that the water absorption increases beyond the precise dosage of 0.10% CO_2 for OPC base mixes and 0.05% CO_2 for PPC-based mixes. The highest increase in water absorption was achieved at 0.20% CO_2 for both OPC and PPC-based mixes. For the OPC-based mixes, an increase of 14-16% in absorption after immersion, up to 20% in absorption after immersion and boiling, and 10-16% in volume of voids are recorded. And for PPC-based mixes, an increase of 13-18% in absorption after immersion, up to 23% in absorption after immersion and boiling, and 12-15% in volume of voids is recorded.

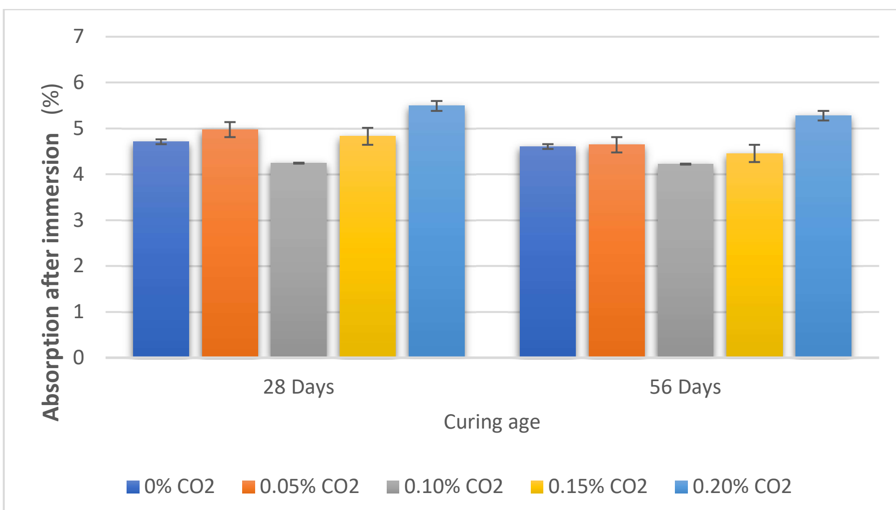


Figure 4. 14 Percentage absorption after immersion (for OPC based mixes)

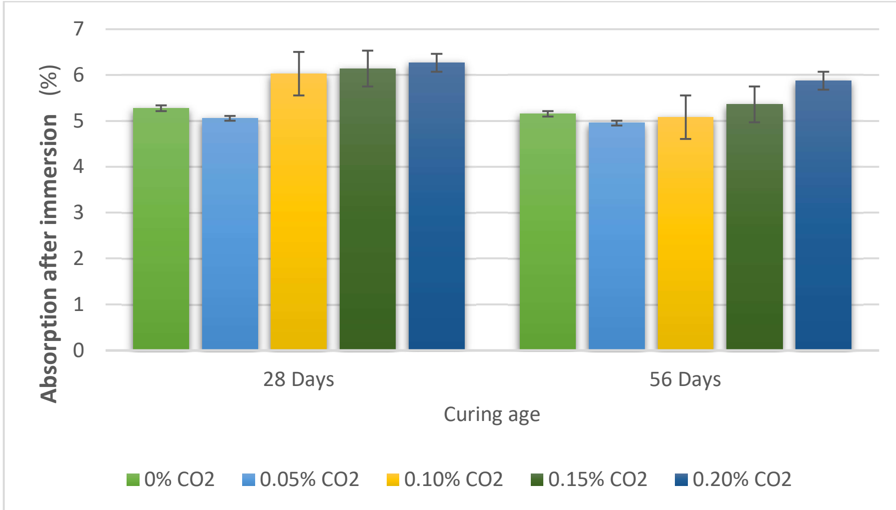


Figure 4. 15 Percentage absorption after immersion (for PPC based mixes)

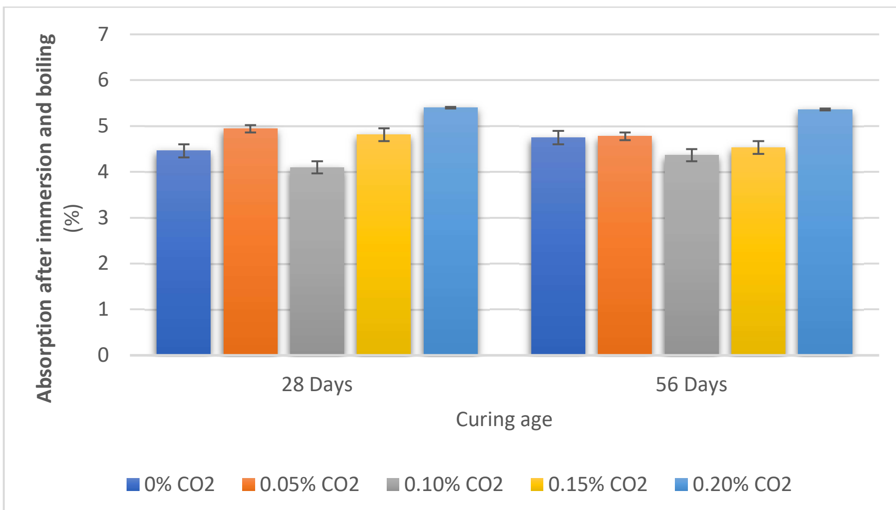


Figure 4. 16 Percentage absorption after immersion and boiling (for OPC based mixes)

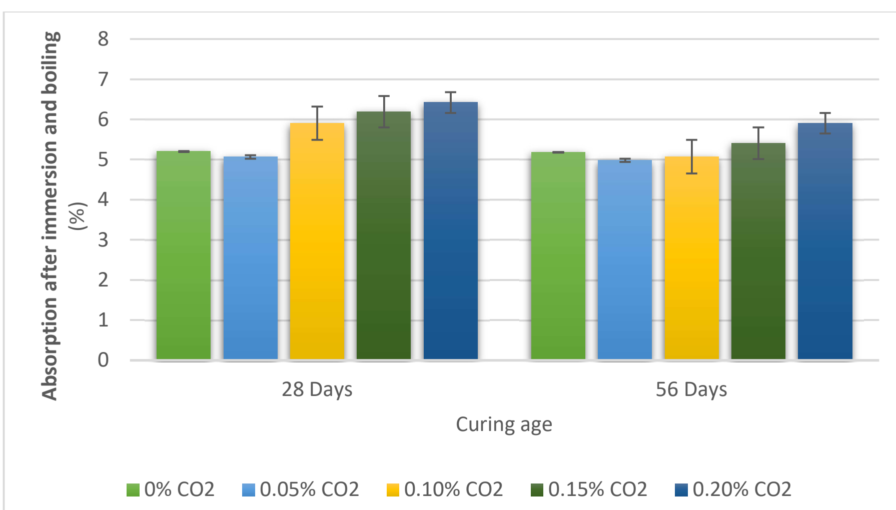


Figure 4. 17 Percentage absorption after immersion and boiling (for PPC based mixes)

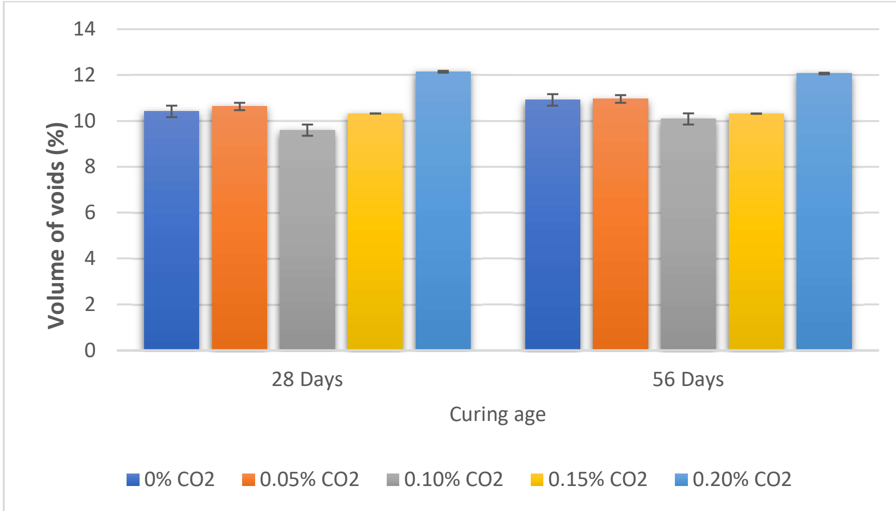


Figure 4. 18 Percentage volume of voids (for OPC based mixes)

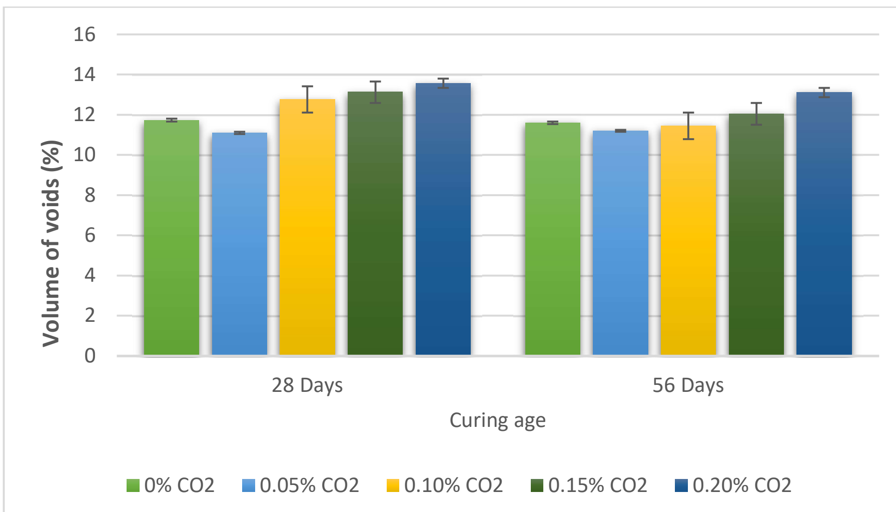


Figure 4. 19 Percentage volume of voids (for PPC based mixes)

4.4.3 Sorptivity

The performance of concrete exposed to extreme environments is a function of the pore system's degree of penetrability. Various variables determine surface absorption in concrete, such as mix proportions, admixture presence, voids, the degree of hydration, type of placement, cementitious material chemical characteristics, etc. Sorptivity is a concrete durability attribute. A material's sorptivity should be as low as feasible to increase its durability. The Table 4.7 depicts the sorptivity coefficients of various mixes with different dosages of carbon dioxide and binder types at 28 and 56 days of curing.

Table 4. 7 Sorptivity coefficient values (mm/s$^{1/2}$)

Mix Type	28 Days		56 Days	
% CO_2	OPC	PPC	OPC	PPC
0	0.0015	0.001	0.0014	0.0004
0.05	0.0014	0.0008	0.0013	0.0003
0.1	0.001	0.0012	0.001	0.0005
0.15	0.0011	0.00125	0.0012	0.0006
0.2	0.0016	0.0013	0.0016	0.00073

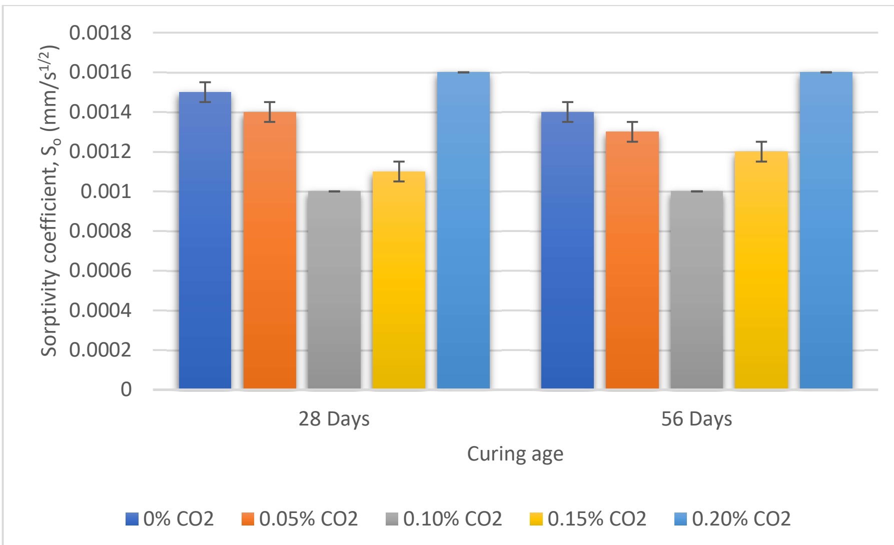


Figure 4. 20 Sorptivity coefficients at 28 and 56 days (for OPC based mixes)

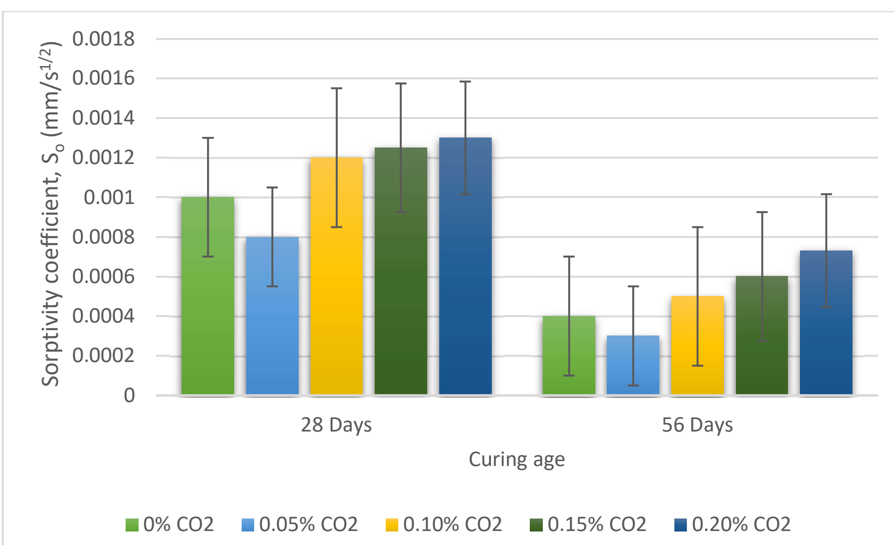


Figure 4. 21 Sorptivity coefficients at 28 and 56 days (for PPC based mixes)

It is evident from Figure 4.20 and Figure 4.21 that the water sorptivity decreases with an increase in CO_2 dosage from 0% to 0.10% for the OPC-based mixes. For PPC-based mixes, water sorptivity decreases for CO_2 dosage from 0% to 0.05%. The least sorptivity coefficient values are observed at 0.10% CO_2 and 0.05% CO_2 across 28 and 56 days of curing for OPC and PPC-based mixes, respectively. The addition of carbon dioxide leads to a decrease in sorptivity. Due to the formation of nano-sized calcium carbonates during carbonation, these calcium carbonates precipitate in the pore spaces to densify the concrete. This densification of concrete decreases the porosity and hence its sorptivity. The sorptivity coefficients for the PPC-based mixes are less than that of OPC-based mixes. It is due to the varying particle size and presence of finer particles than OPC that fill up the voids and reduces their porosity. The major difference in the sorptivity values for PPC-based mixes can be observed at 56 days of curing. The 38% reduction in the sorptivity coefficient for a 0.05% CO_2 mix can be seen at 56 days of curing (see Figure 4.21).

Figure 4.22 to Figure 4.25 depicts the sorptivity absorption curves for different concrete mixes with varying dosages of carbon dioxide and binder types at 28 and 56 days of curing. Both initial and secondary absorption curves are drawn for different concrete mixes. A similar trend to the sorptivity coefficient values can be seen.

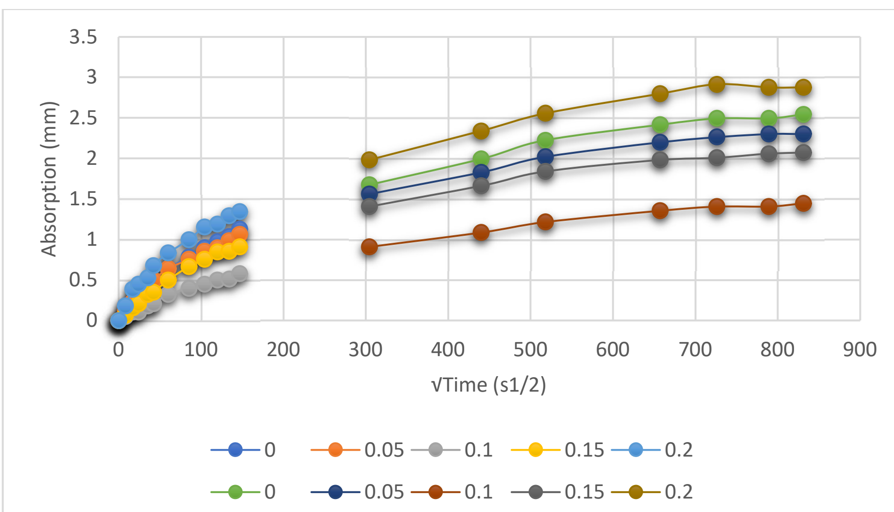


Figure 4. 22 Sorptivity test results at 28 days (for OPC based mixes)

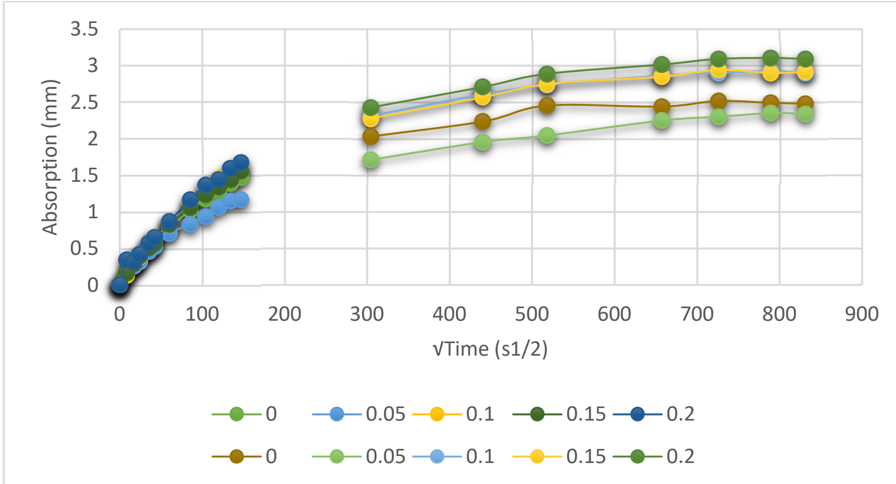


Figure 4. 23 Sorptivity test results at 28 days (for PPC based mixes)

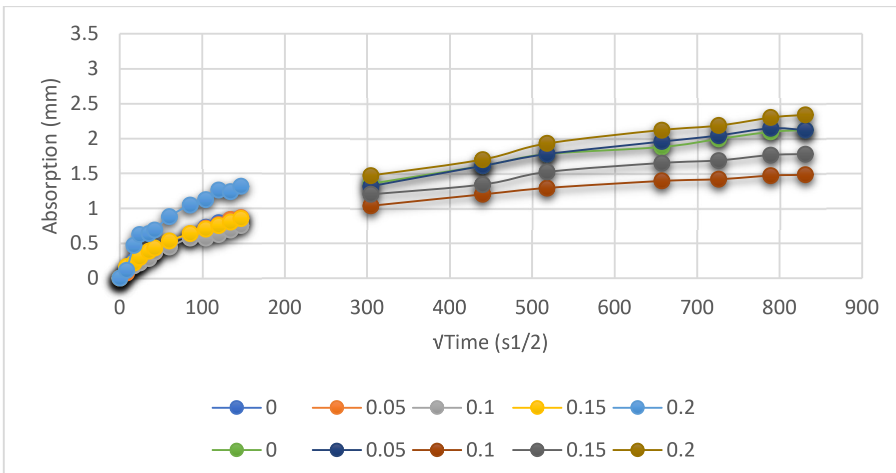


Figure 4. 24 Sorptivity test results at 56 days (for OPC based mixes)

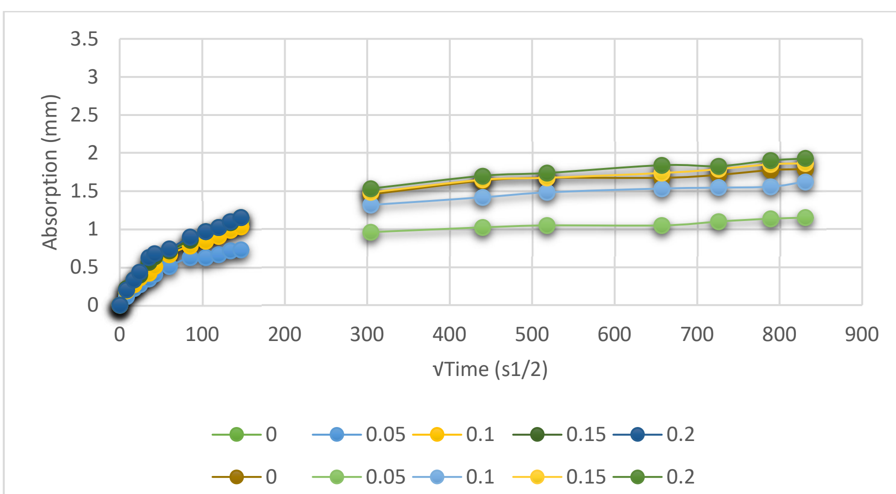


Figure 4. 25 Sorptivity test results at 56 days (for PPC based mixes)

4.4.4 pH

The carbon dioxide in concrete is said to be a durability issue. One of the most significant factors responsible for reducing pH value in the concrete is carbon dioxide, like atmospheric carbonation. However, adding carbon dioxide to the concrete during mixing might raise some concerns. Thus, to rectify these concerns, the pH of the hardened concrete was investigated at various ages. Given the importance of pH for durability, the pH was analyzed by three different methods.

1. **By pH paper:** The core of the split concrete cylindrical specimens from the tensile strength test were sprayed with distilled water, and the pH paper then determined the pH of the sprayed surface. The pH paper turned greyish for all the specimens indicating the pH range of 12 to 14.

Figure 4. 26 pH paper test results for all mixes

2. **By pH meter:** The precise pH values were obtained using the pH meter. The pH meter was dipped into the filtered solution obtained from powdered concrete and distilled water. Table 4.8 presents the pH values of the various concrete mixes at different ages.

Table 4. 8 pH values for all mixes at 7, 28, and 56 days

Mix Type	**7 Days**		**28 Days**		**56 Days**	
% CO_2	OPC	PPC	OPC	PPC	OPC	PPC
0	12.27	12.17	12.36	12.19	12.36	12.22
0.05	12.26	12.22	12.39	12.26	12.38	12.28
0.1	12.31	12.21	12.36	12.28	12.38	12.24
0.15	12.26	12.26	12.38	12.29	12.42	12.27
0.2	12.35	12.29	12.36	12.53	12.36	12.31

3. **By phenolphthalein indicator:** The core of the split concrete cylindrical specimens from the tensile strength test were sprayed with the phenolphthalein indicator. Upon spraying by the phenolphthalein indicator, all the specimens turned pinkish (as shown in Figures 4.27 to 4.30), indicating the pH of basic nature.

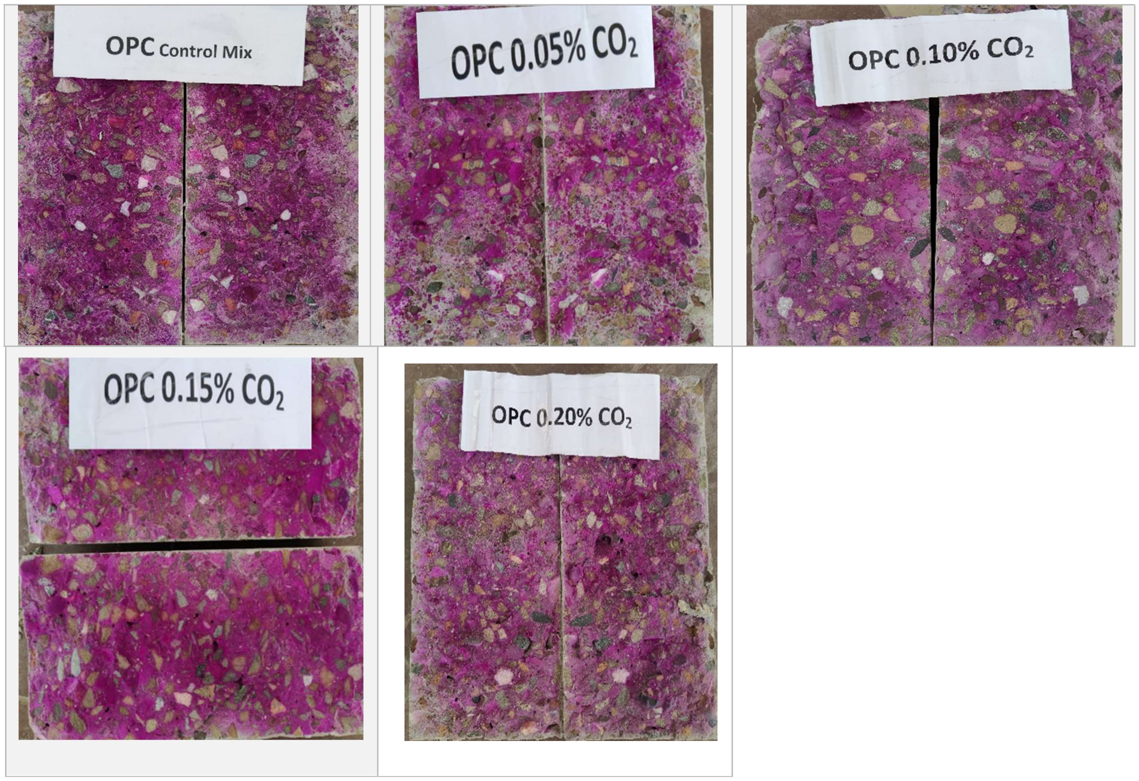


Figure 4. 27 Phenolphthalein sprayed specimens at 28 days (OPC based mixes)

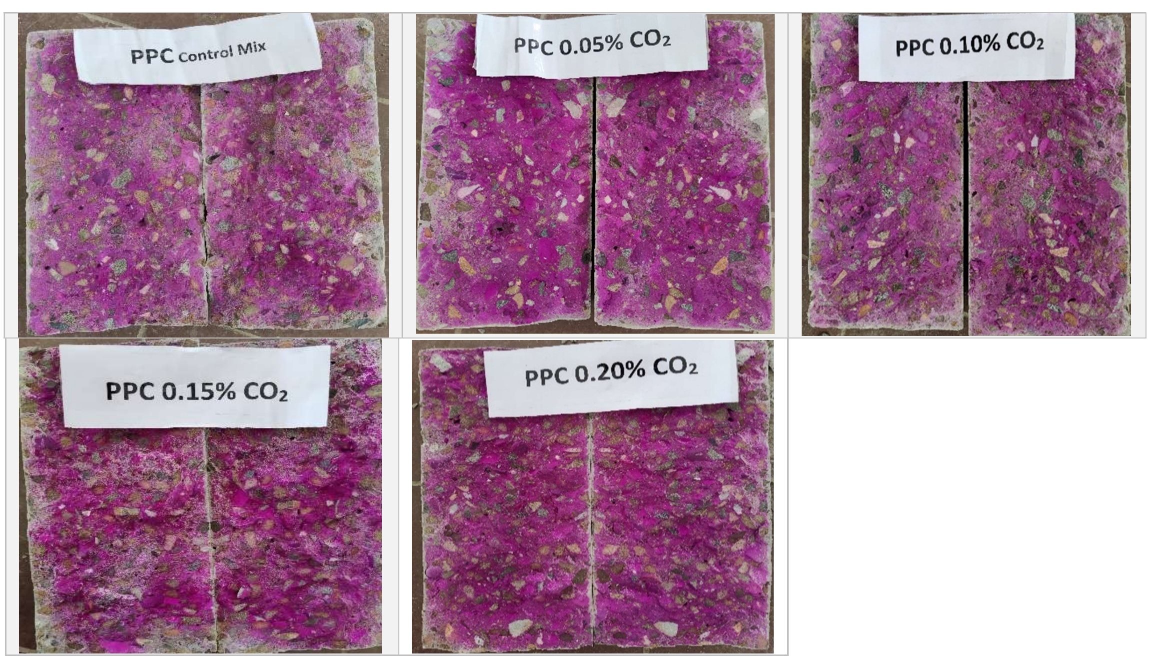


Figure 4. 28 Phenolphthalein sprayed specimens at 28 days (PPC based mixes)

Figure 4. 29 Phenolphthalein sprayed specimens at 56 days (OPC based mixes)

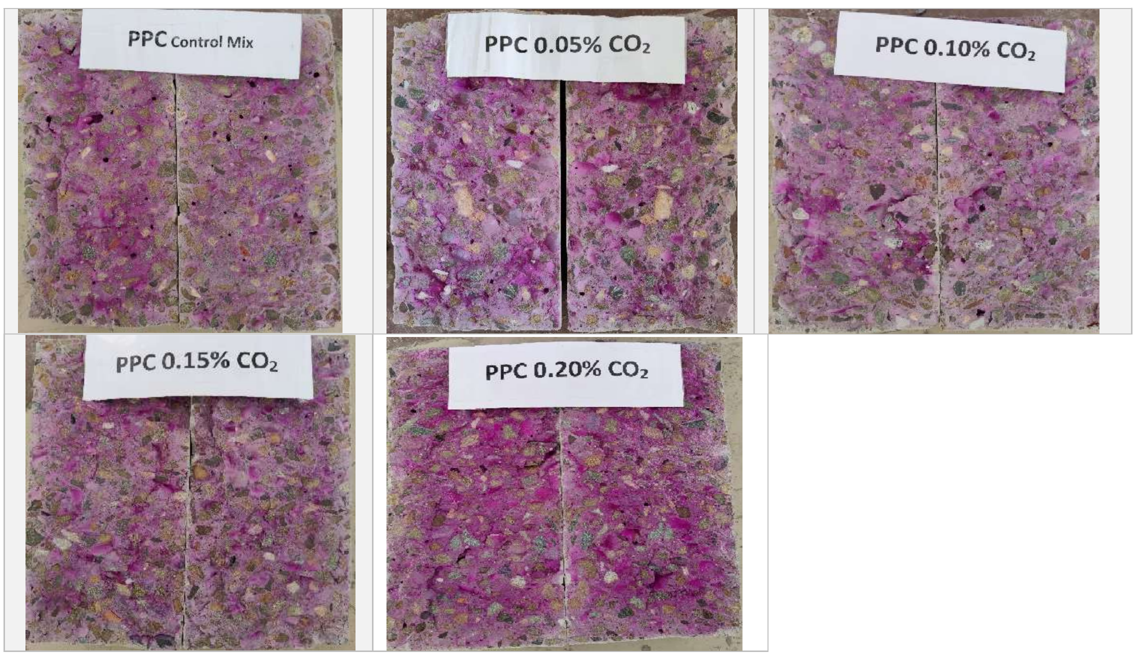


Figure 4. 30 Phenolphthalein sprayed specimens at 56 days (PPC based mixes)

Each test result showed that the carbon dioxide injection had no detrimental influence on the pH of the concrete. Thus, it can be concluded that the addition of carbon dioxide during mixing did not have any adverse effect on the alkalinity of the concrete, making it feasible for the utilization in the reinforced concrete as well.

4.5 Non-Destructive Tests

The results acquired from the rebound hammer test and UPV with their respective compressive strength results are depicted in Table 4.9.

Table 4. 9 Non-Destructive test results with respective compressive strengths

OPC			
CO_2 (%)	**Compressive strength**	**Rebound hammer**	**UPV**
0	37.625	35.135	4531.667
0.05	39	36.278	4533.333
0.10	41.25	38.854	4535
0.15	36.875	33.683	4490
0.20	35	31.587	4453.333
PPC			
0	41.8333	33.333	4528.333
0.05	49.923	36.833	4548.333
0.10	44.2083	35.266	4531.667
0.15	39.519	32.158	4510
0.20	35.0416	30.782	4490

4.5.1 Ultrasonic pulse velocity (UPV)

The addition of carbon dioxide to the concrete did not significantly affect the values of the ultrasonic pulse velocity. Overall velocity values observed are in the good or excellent category range, showing that concrete quality is not affected much by adding carbon dioxide. It is observed from the Figure 4.31 that with the precise dosage of 0.10% CO_2 for OPC and 0.05% CO_2 for PPC, there is a slight increase in the velocity. But beyond the precise dosage, the velocity tends to decrease slightly. The mix with 0.20% CO_2 for both OPC and PPC-based mixes had the least velocity among all.

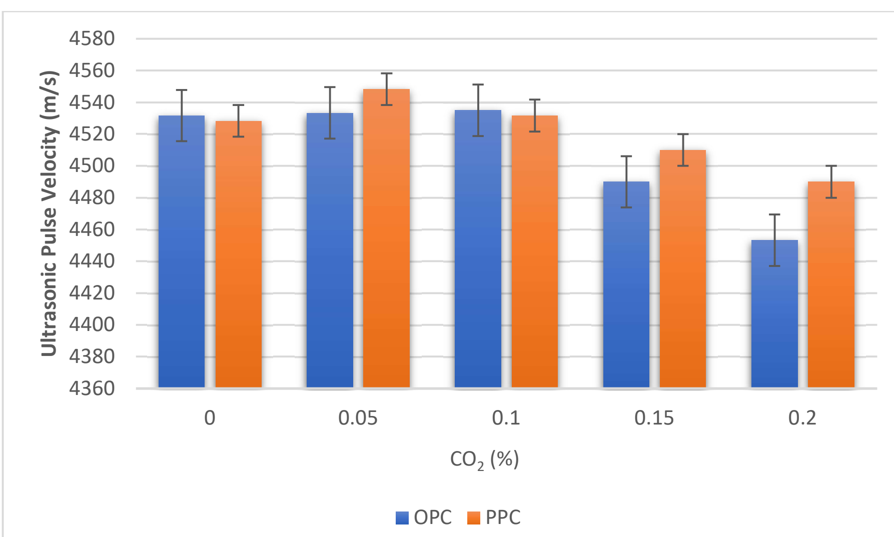


Figure 4. 31 UPV values vs CO_2 percentage at 28 days

Figure 4.32 shows the best fit plot between compressive strength and the ultrasonic pulse velocity values at 28 days of curing, and the Equations below show the relationship.

CS = 0.2414UPV - 1049.6 (for PPC based mixes)

CS = 0.0551UPV - 210.34 (for OPC based mixes)

Where CS is compressive strength in N/mm^2 and UPV is UPV values in m/s. By performing UPV on the casted concrete, the compressive strength of the carbonated concrete can be estimated so that a destructive test need not be performed.

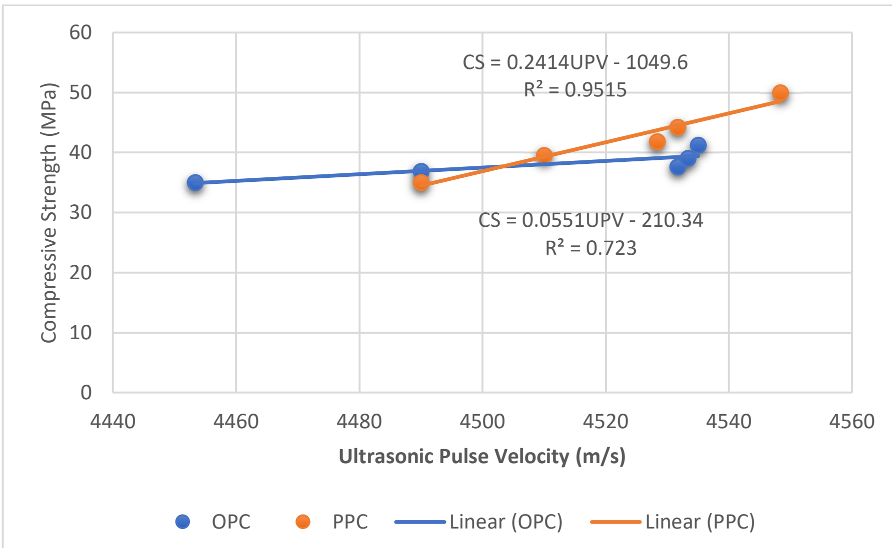


Figure 4. 32 UPV values vs Compressive strength results

4.5.2 Rebound Hammer

The compressive strength by rebound hammer test of the all-concrete mixes shows a decrement compared to the experimental Compressive Strengths. This behaviour may be due to the application of only a point load on the specimens in the rebound hammer test. At the same time, the uniform loading is applied in the CTM for calculating the experimental Compressive Strength. It is observed from Figure 4.33 that with the precise dosage of 0.10% CO_2 for OPC and 0.05% CO_2 for PPC, there is a slight increase in the compressive strength by rebound hammer test. But beyond the precise dosage, the strength tends to decrease slightly. The mix with 0.20% CO_2 for both OPC and PPC-based mixes had the least strength among all.

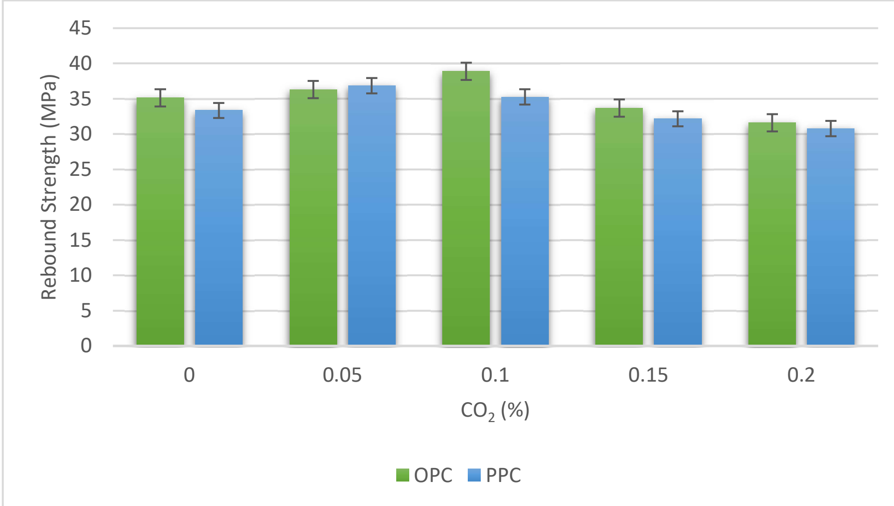


Figure 4. 33 Rebound hammer values vs CO_2 percentage at 28 days

Figure 4.34 shows the best fit plot between rebound hammer values and their corresponding destructive compressive strengths. The equation gives a relationship between rebound strength and compressing strength of various mixes with different dosages of carbon dioxide.

CS =2.2534Rb - 33.779 (for PPC based mixes)

CS = 0.8548Rb + 7.9404 (for OPC based mixes)

Where CS is the destructive compressive strength, and Rb is the corresponding rebound strength for the cubes. The equation would help estimate the compressive strength of concrete using a rebound hammer.

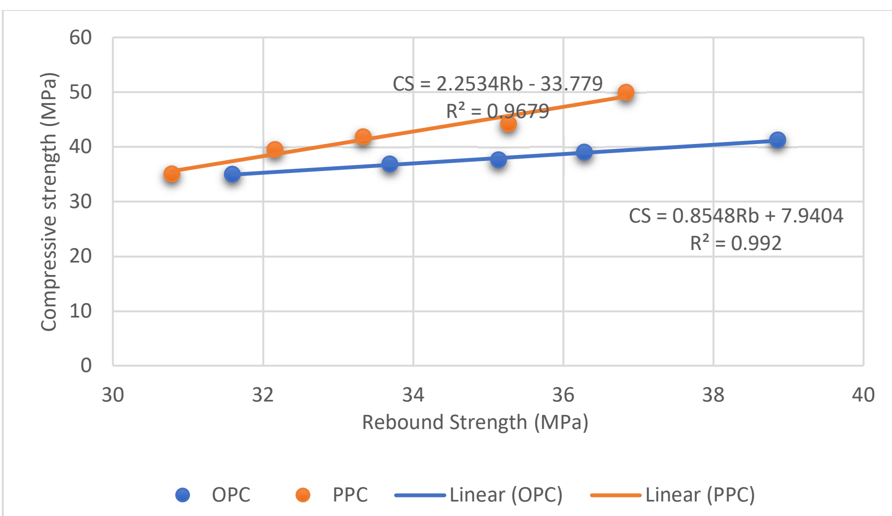


Figure 4. 34 Rebound strength vs Compressive strength results

4.6 Microstructural analysis

4.6.1 X-Ray diffraction analysis

Qualitative analysis shows the presence of Gismondine (G), Calcite/Calcium carbonate (C), Quartz (Q), and Portlandite/Calcium hydroxide (P) as major crystalline hydrated phases in both carbonated and non-carbonated samples of OPC and PPC based mixes. Figure 4.35 and Figure 4.36 show the XRD analysis of OPC and PPC-based mixes at 56 days, respectively. The predominant element in the carbonated samples was calcium carbonate polymorphs, represented cumulatively as calcium carbonate.

It is apparent from the results that all the hydrated phases get carbonated simultaneously upon exposure to carbon dioxide. It can also be observed that no significant phase change was detected upon the addition of carbon dioxide. It is also evident that the peaks of calcite are more prominent in carbonated samples than that of non-carbonated samples or other elements, indicating carbonation of the amorphous phase (basically, the reactions mentioned in 1.6 has taken place). A clearer picture could have been seen in the quantitative analysis, which was out of the scope of work. However, SEM images also correlate with the results of XRD analysis, showing a more homogeneous mix and calcite ($CaCO_3$) presence in carbonated samples. However, XRD cannot characterize the effect of CO_2 addition on the cement hydration process product; the hydrated product's diffraction peak increases with the increase of CO_2 dosages. The diffraction peak of Calcite also increases with the increase of CO_2 dosage, which indicates that the incorporation of CO_2 improves the hydration of cement.

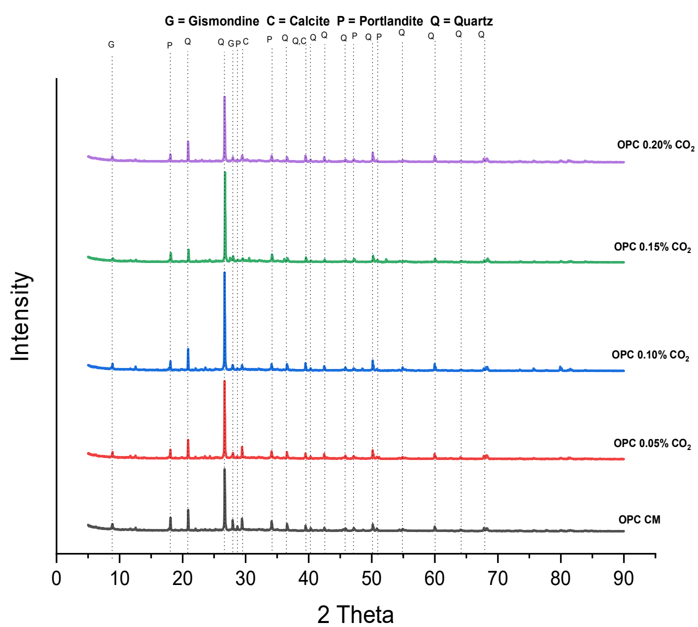


Figure 4. 35 X-ray diffraction patterns at 28 days for OPC-based mixes

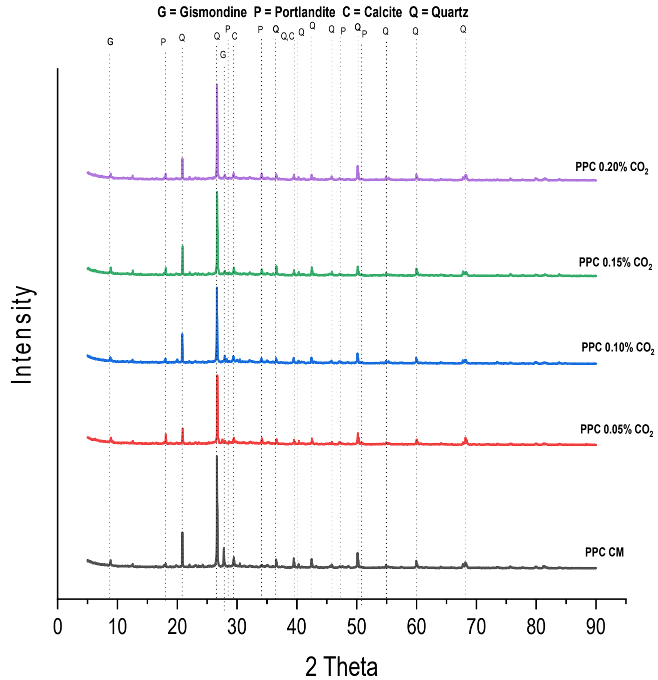


Figure 4. 36 X-ray diffraction patterns at 28 days for PPC-based mixes

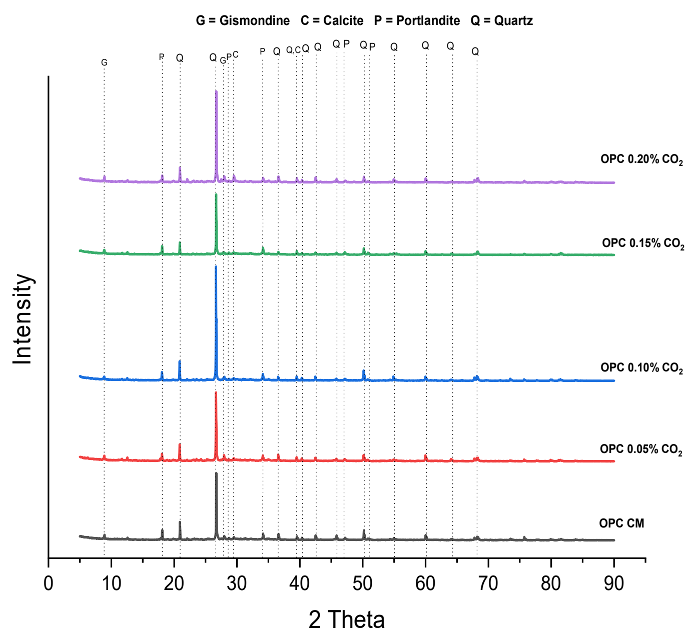


Figure 4. 37 X-ray diffraction patterns at 56 days for OPC-based mixes

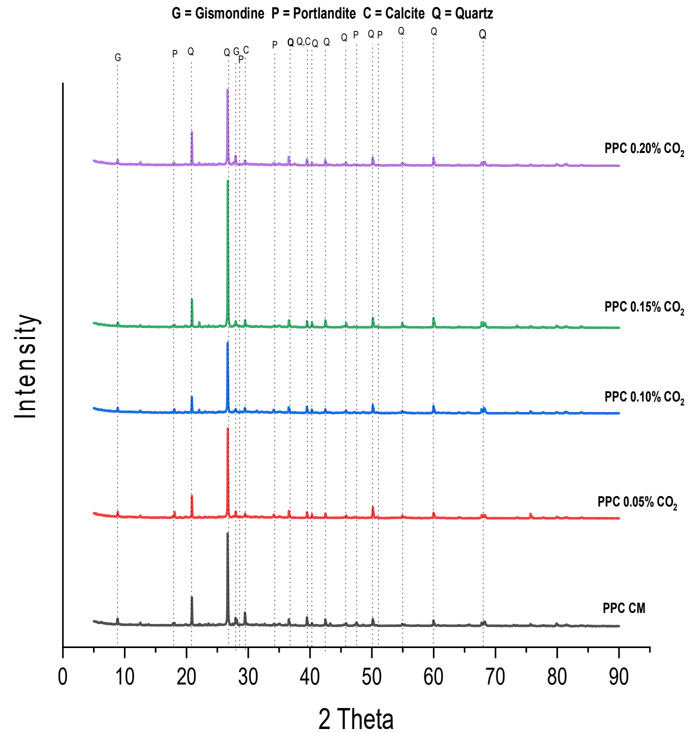
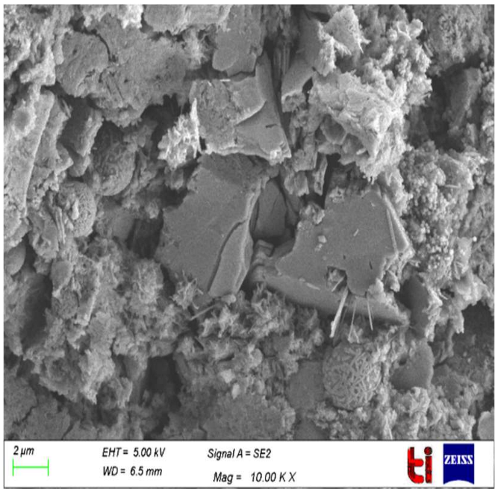


Figure 4. 38 X-ray diffraction patterns at 56 days for PPC-based mixes

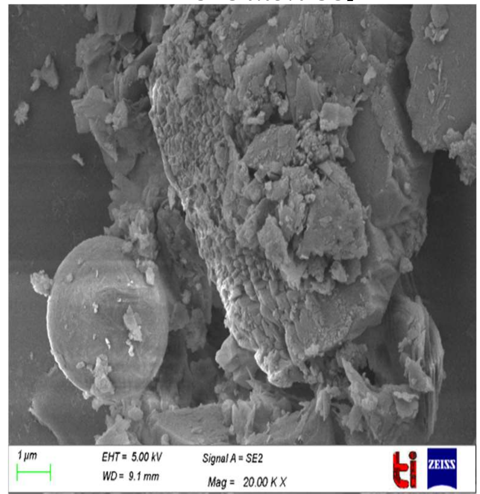
4.6.2 SEM of broken concrete samples

SEM micrographs of broken concrete samples in Figure 4.37 to Figure 4.40 show carbon dioxide injected concrete specimens are denser and less porous than control specimens. It can also be seen that carbon dioxide changes the morphology of the concrete, like in OPC for 0.10% CO_2 and in PPC for 0.05% CO_2. Clearly, lesser ettringite and more rod-like dense hydration products can be seen. The dense and more compact microstructure is observed for the OPC-based mix at 0.10% CO_2 dosage, and for the PPC-based mix at 0.05% CO_2 dosage, the capillary pores observed at these dosages are almost negligible, which is also in coherence with the results of compressive strength and is also evident in durability properties. It is concluded that the addition of carbon dioxide can change the hydration process of the cement.

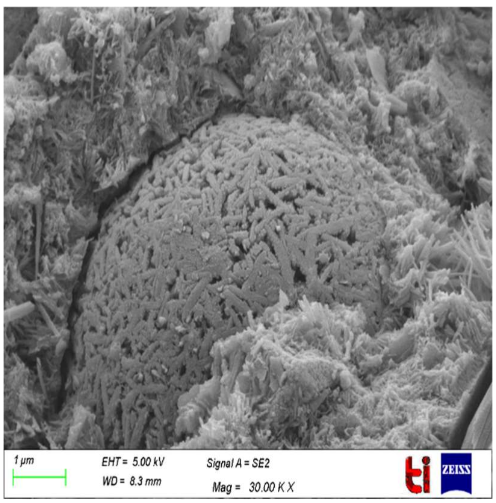
OPC CM



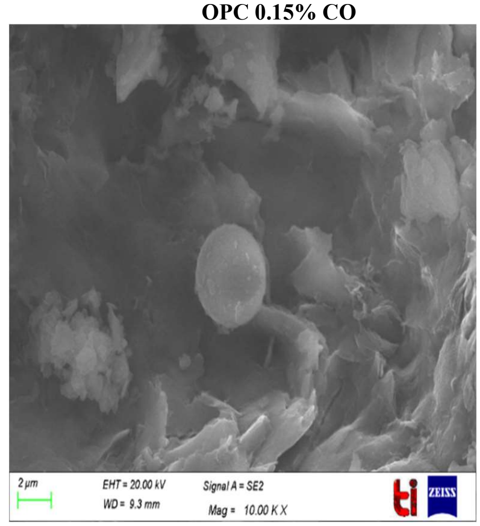
OPC 0.05% CO_2



OPC 0.10% CO_2



OPC 0.15% CO



OPC 0.20% CO_2

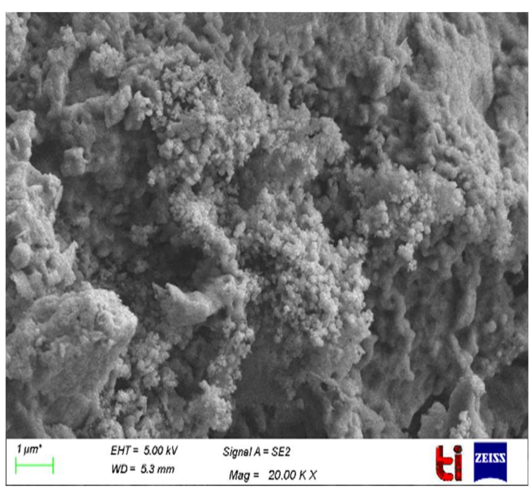
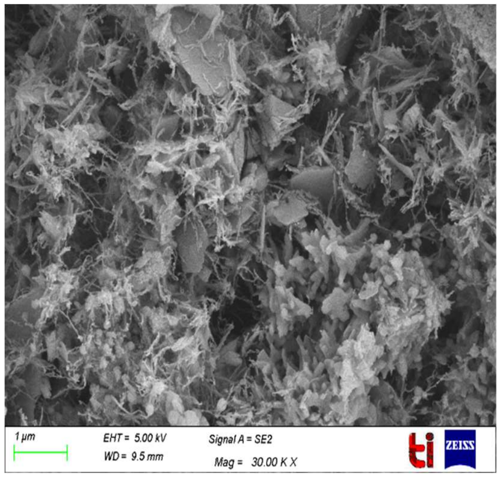
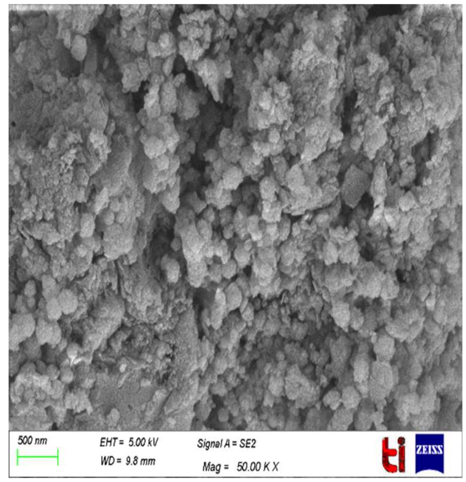


Figure 4. 39 SEM images of OPC based mixes at 28 days

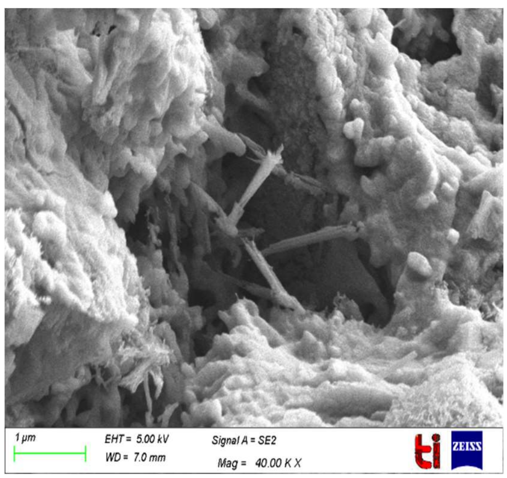
PPC CM



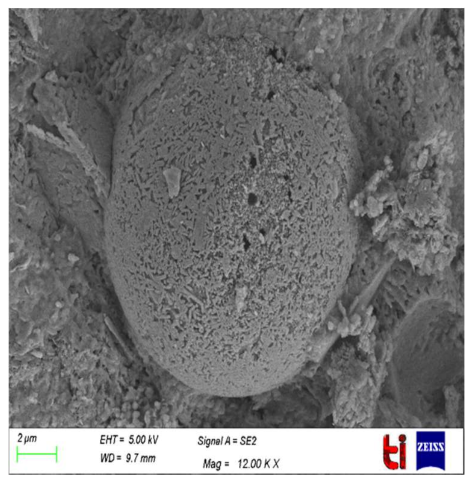
PPC 0.05% CO_2



PPC 0.10% CO_2



PPC 0.15% CO_2



PPC 0.20% CO_2

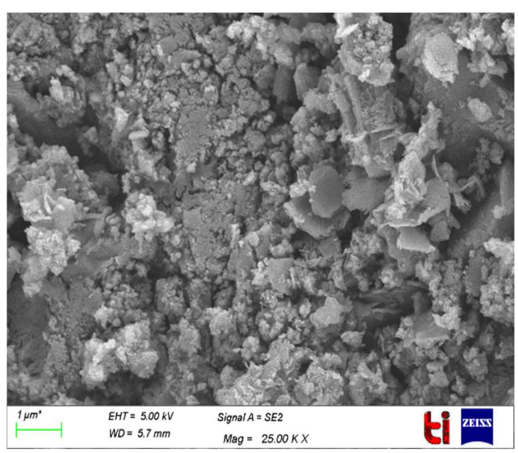


Figure 4. 40 SEM images of PPC based mixes at 28 days

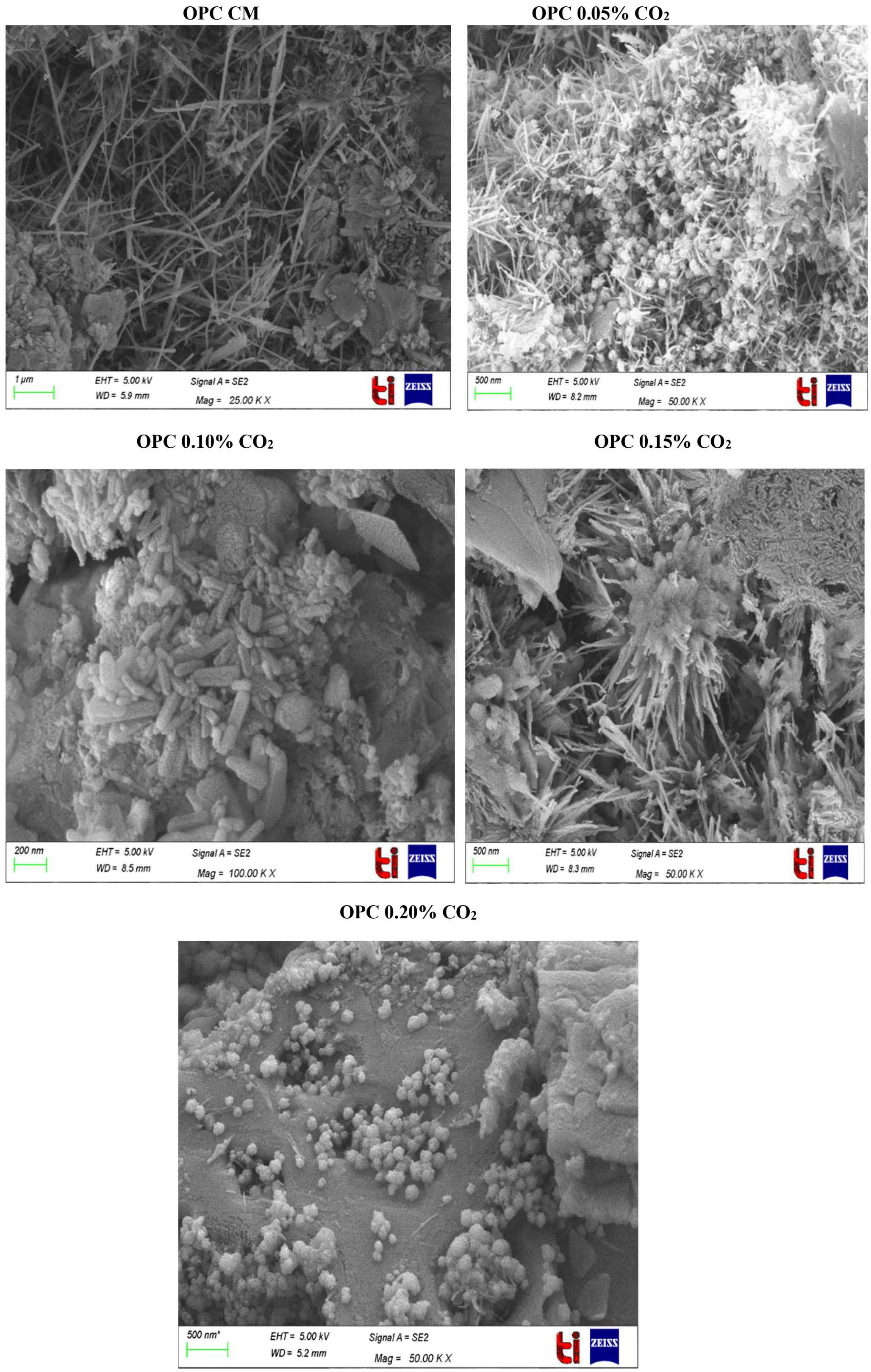
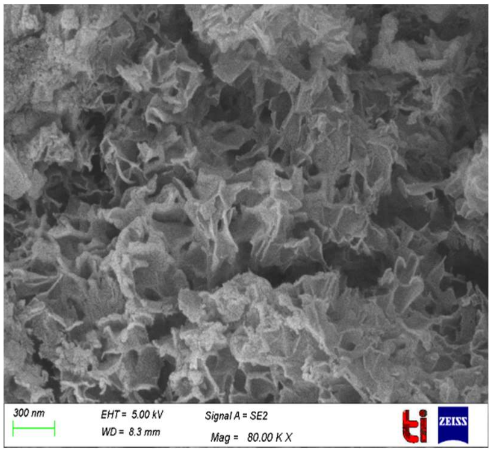
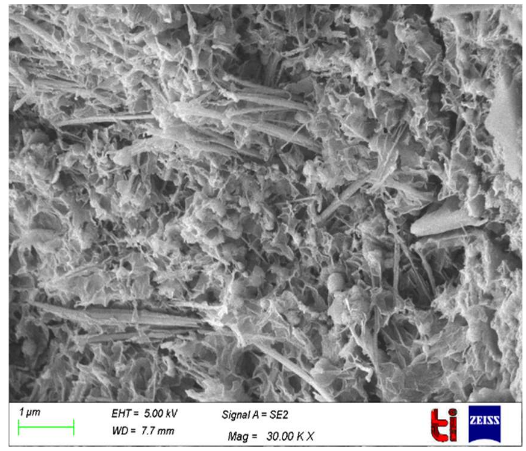


Figure 4. 41 SEM images of OPC based mixes at 56 days

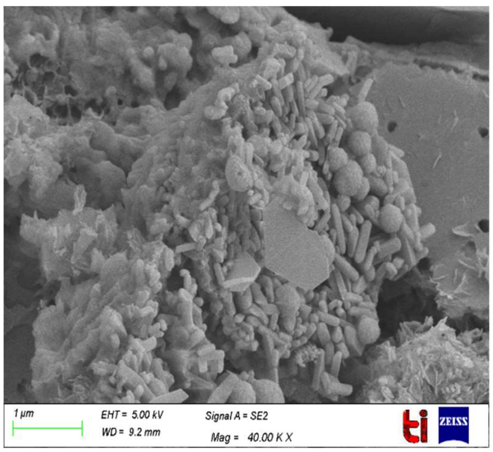
PPC CM



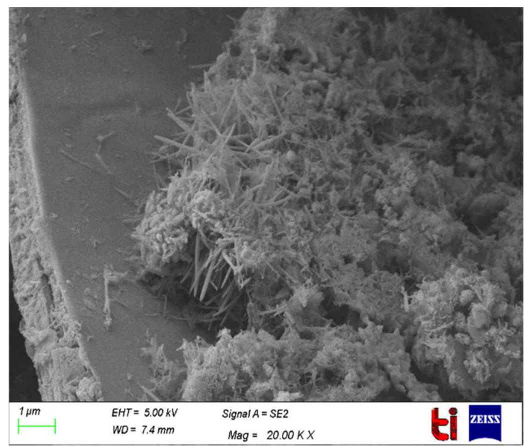
PPC 0.05% CO_2



PPC 0.10% CO_2



PPC 0.15% CO_2



PPC 0.20% CO_2

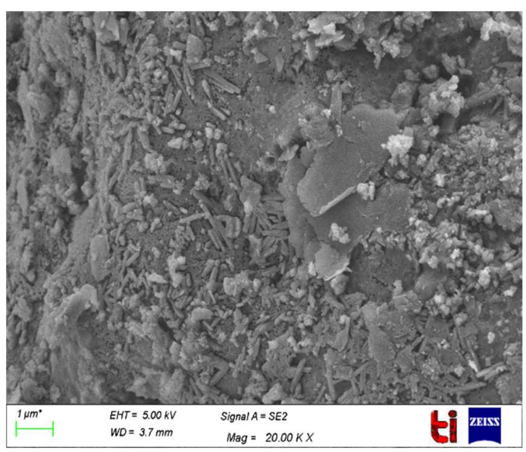


Figure 4. 42 SEM images of PPC based mixes at 56 days

CHAPTER 5

Conclusions

The effect of carbon dioxide addition on the different characteristics of concrete was studied at dosages of 0%, 0.05%, 0.10%, 0.15%, and 0.20% by weight of the binder. The following are the main findings of this study:

1. Carbon dioxide has no adverse influence on the properties of fresh concrete. All of the properties were deemed acceptable. The addition of carbon dioxide caused minor changes in the fresh properties, and the carbon dioxide-treated concrete samples were evaluated to perform comparably to the control mix.
2. The PPC-based mixes were less workable as compared to that of OPC-based mixes. Thus, to increase the workability of the PPC-based mixes, a more superplasticizer is required.
3. Carbon dioxide injection into concrete mixes enhanced setting and strength development without impairing fresh properties. For OPC-based mixes, the time to the initial set was reduced by 75 minutes, and the time to the final set was reduced by 103 minutes. The time to the initial set for PPC-based mixes was accelerated by 85 minutes, while the time to the final set was accelerated by 127 minutes.
4. The concrete mixtures that were treated with carbon dioxide showed an increase in compressive strength. For OPC-based mixes, concrete samples with 0.10% CO_2 showed a compressive strength benefit of up to 30%, whereas, for PPC-based mixes, concrete samples with 0.05% CO_2 showed a compressive strength benefit of up to 25%. Furthermore, PPC-based concrete samples had the highest compressive strengths at later ages, whereas OPC-based concrete samples had the highest compressive strengths at early ages.
5. The CO_2-injection technique had a neutral to positive impact on the concrete's durability, according to the durability tests. Testing confirmed the CO_2-treated concrete's performance in terms of chloride penetration resistance, pH, and water absorption resistance. The PPC-based concrete samples outperformed the OPC-based samples in terms of resistance to water absorption and chloride penetration.
6. The carbon dioxide in the concrete is thought to lower the pH of the concrete. However, there were no such problems with the process of adding carbon dioxide while mixing. Concrete's pH was assessed at various ages. The results indicated that there was no discernible change in the pH levels. This demonstrates the prior claim that adding carbon dioxide during mixing has no impact on the pH of the concrete.

7. Carbon dioxide might be a low-cost and viable accelerator. Carbon dioxide addition may be utilized to improve a treated mix and seek binder blends with lower carbon intensities.
8. The microstructural investigations revealed that the carbon dioxide addition could change the morphology and optimize the hardened structure of the concrete.

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