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Original Article

CO_2-Based Tile Curing: A Comprehensive Experimental Assessment of Performance

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Abstract - *This study evaluates the performance of CO_2-cured tiles as an environmentally friendly construction material, focusing on the early curing stages at 3 days and 7 days. The research aims to reduce water usage during tile production and assess CO_2 sequestration into the tiles after controlled CO_2 curing, quantifying CO_2 absorption. Comprehensive experiments were conducted to analyze the mechanical and physical properties of CO_2-cured tiles at 3 days and 7 days. The study examined compressive strength, water absorption, and dimensional stability. Water usage was optimized in the production process, and advanced techniques were used for qualitative analysis of CO_2 sequestration. CO_2-cured tiles exhibited favorable compressive strength development at both early curing stages, demonstrating durability. Water absorption was significantly reduced, aligning with the goal of minimizing water usage. Qualitative analysis confirmed successful CO_2 sequestration within the tiles, and BET analysis quantified their CO_2 absorption capacity. CO_2-cured tiles show promise for sustainable construction, with strong early-stage performance, reduced water usage, and effective CO_2 sequestration. These findings support the use of CO_2-cured tiles as a solution for reducing carbon emissions and promoting sustainable building practices.*

Keywords - *CO_2-cured tiles, Early-age performance, Water usage reduction, CO_2 sequestration, BET analysis, Carbon emissions reduction.*

1. Introduction

The environmental pollution lies in embracing new energy efficiencies and CO_2 emission reduction technologies. By doing so, we can combat climate change, protect the environment, and secure a more sustainable and prosperous future for humanity [1]. However, achieving these goals will demand collective efforts and a commitment to innovation, investment, and global collaboration. The content discusses tile manufacturing as a highly energy-intensive process that emits carbon dioxide (CO_2).

Tile production involves several stages requiring thermal treatment, contributing significantly to CO_2 emissions. As a result, tile manufacturing has become one of the main sources of carbon dioxide emissions globally. In recent years, CO_2 emissions from tile manufacturing have reached a substantial figure of 100 million metric tons [2]. This highlights the importance of finding more sustainable and eco-friendly methods for tile production to mitigate the environmental impact of this industry.

The method of accelerated curing for tiles involves the diffusion of carbon dioxide (CO_2) into the tile by controlling specific pressure and temperature conditions. This process facilitates the CO_2 gas to permeate the tile, initiating a carbonation process [3]. Carbonation is the reaction of CO_2 with certain materials, such as cement, to form carbonate compounds, which are likely used in the production of tiles and are a major contributor to global emissions. Specifically, cement production accounts for nearly 5% of the total global emissions of greenhouse gases, particularly CO_2.

The use of accelerated curing by carbon dioxide diffusion as a method in tile manufacturing also underscores the significant impact of cement production on global CO_2 emissions [4]. It implies that finding sustainable and eco-friendly methods for cement and tile production could help reduce their environmental footprint.

The process of carbon dioxide diffusion into tiles is conducted within a controlled environmental condition in a chamber, referred to as the “carbonation curing process.” This method aids in achieving early strength in the tiles while also contributing to reducing environmental pollution, presumably by sequestering CO_2 into the tiles.

The concept of “carbon credit” or “carbon offset.” A carbon credit is a unit that represents the reduction or removal of greenhouse gas emissions from the atmosphere through emission reduction projects. Governments, industries, or individuals can use these carbon credits to compensate for the emissions they produce. Essentially, it allows entities to offset their carbon footprints by investing in or supporting initiatives that reduce greenhouse gas emissions elsewhere, effectively balancing out their emissions. The carbonation curing process in tile manufacturing could potentially lead to the creation of carbon credits, as it helps reduce CO_2 emissions. These carbon credits can be valuable in environmental and sustainability initiatives and incentivize industries to adopt more eco-friendly practices [5].

1.1. Importance of CO_2 Cured Samples

1.1.1. Performance of CO_2-cured Sample at Early Ages

The project aims to assess the performance of the tiles after being cured with CO_2 for 3 days and 7 days [6]. This will help determine the strength and other properties of the tiles at these early stages of curing.

1.1.2. Reducing Water Usage

One goal is to minimize water consumption in the Sample manufacturing process. This is likely to contribute to overall sustainability and resource conservation.

1.1.3. Qualitative Analysis of CO_2 Sequestration

The project seeks to qualitatively analyze how effectively the CO_2 gas is absorbed and sequestered into the sample during the carbonation curing process in the chamber [7]

1.1.4. Quantitative Analysis Using BET Analysis

The project aims to quantitatively estimate the amount of CO2 absorbed into the sample using the BET analysis method to complement the qualitative analysis. BET analysis is a technique commonly used to determine the surface area and adsorption capacity of materials [8]

1.1.5. Reducing Carbon Emissions Through CO2 Sequestration

The project aims to reduce carbon emissions by effectively sequestering CO_2 gas into the sample [9]. This means the sample would act as a carbon sink, helping offset or neutralize CO_2 emissions from other sources.

2. Material and Methods

2.1. Materials with Sample Preparation

2.1.1. Materials Used

M/S Carbon Craft has provided the laboratory in GITAM with the necessary materials for preparing the samples [10]. This includes two bags of the top layer sample mixture containing carbon, marble chips, and marble powder. Two bags of the bottom layer sample mixture are supplied, consisting of carbon, gravel powder, stone dust, and 6mm hard stone gravel. The laboratory also received one mould for sample casting.

2.1.2. Local Procurement of Cement

While most of the materials are supplied by M/S Carbon Craft, the laboratory has locally procured the cement required for the tile preparation. Cement is a key component in the tile manufacturing process.

The preparation of the samples will likely involve mixing the provided materials in the specified proportions and using the mould to shape and cast the tiles [10]. Depending on the specific methodology suggested by M/S Carbon Craft, additional steps like curing and drying may also be involved to complete the sample preparation process. Once the samples are prepared, they can be subjected to the carbonation curing process and undergo testing to assess their transverse strength and the amount of carbon dioxide absorbed, as mentioned in the previous description of the project objectives [11].

Fig. 1 Top layer sample mixture

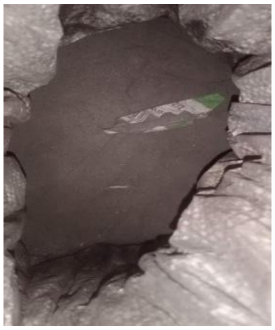


Fig. 2 Bottom layer sample mixture

2.2. The Research Progress

2.2.1. Casting and Methodology

The samples were cast using materials provided by the client, following the established manufacturing unit’s procedures [12].

2.2.2. Curing Process

The curing process involved the introduction of carbon dioxide into the sample under specific pressure and temperature conditions for a duration of 4 hours.

2.2.3. Transverse Strength Testing

Following the carbonation curing process, the CO_2-cured samples were air-dried for 3 days and 7 days before conducting transverse strength tests at these time points.

2.2.4. Comparison with Other Curing Methods

To assess the effectiveness of CO_2 curing, the transverse strength of CO_2-cured samples was compared with the sample cured using water alone and the sample left to cure in the open air, both tested at the same ages (3 days and 7 days).

2.2.5. Qualitative and Quantitative Analysis

Qualitative tests were performed on the CO_2-cured samples to confirm the presence of carbon dioxide within them. Additionally, quantitative tests were conducted to estimate the amount of CO_2 absorbed by the sample during the curing process [13]. This comprehensive evaluation of the CO_2 curing process for samples involves comparing it to conventional curing methods. Testing transverse strength and qualitative and quantitative analyses are essential steps in assessing the feasibility and efficiency of CO_2 curing in terms of sample performance and carbon sequestration capabilities [14].

2.3. The Technical Details of the Double Body Solid Color Terrazzo Tile[1]

Tile Type	Double body solid color terrazzo tile.
Size of the Tile	150mm X 150mm**.**
Weight	1-1.2 kg.
Thickness	18-20mm**.**
Top Layer Thickness	8-9 mm.
Bottom Layer Thickness	10-11 mm.
Ingredients of the Top Layer	Carbon, Cement, Gravel powder, Stone dust, 6mm hard stone gravel.
Ingredients of the Bottom Layer	Carbon, Marble chips (2-3mm), Marble powder, Cement**.**

3. Results and Discussions

The study has conducted tests to determine the casting of the sample and flexural strength of the sample exposed to carbon dioxide (CO_2) along with air curing, as well as their companion sample cured in air and water. Additionally, the presence of carbon dioxide in the sample has been confirmed using four different tests: Lime Water Test, Bromothymol Blue Indicator Test, Liquid Ammonia Test, and Phenolphthalein Indicator Test.

3.1. Casting of Tiles

The tiles' casting procedure follows the guidelines specified in IS 1237(2012). The steps are outlined below:

3.1.1. Top Layer Preparation

The top layer of the sample is already dry mixed with the other materials, which include carbon, cement, gravel powder, stone dust, and 6mm hard stone gravel.

3.1.2. Bottom Layer Preparation

Prepare the bottom layer mixture by mixing three parts of the bottom layer material with one part of cement. The bottom layer comprises carbon, marble chips (2-3mm), marble powder, and cement.

3.1.3. Check Water Consistency

Ensure that the water consistency in the mixture is appropriate. The correct amount of water is essential for achieving the desired properties and workability of the sample mixture.

3.1.4. Apply Grease/Oil

Grease or oil should be applied to the edges of the tile mould to prevent the sample from sticking to the mould during the casting process.

3.1.5. Place Bottom Layer

Pour the prepared bottom layer mixture into the mould first, filling it to a depth of around 10mm.

3.1.6. Place Top Layer

On top of the bottom layer, add the dry mixed top layer mixture up to a depth of approximately 10mm. Once added, compress the top layer mixture to ensure proper bonding and consolidation.

3.1.7. Demoulding

Allow the tile to cure and set in the mould for the required duration. After the curing period is complete, carefully demould the tile from the mould.

The tiles are now ready for further processing, which includes the carbonation curing process and subsequent testing, as explained in the previous parts of the content. This casting procedure is crucial in obtaining the desired composition and structure of the double-body solid color terrazzo tiles.

Fig. 3 Mould for the tile casting

3.2. Procedure for Curing of Samples

The investigation compares the efficacy of tiles cured or dried using traditional curing in water, curing in air alone, and a new method involving diffusion of carbon dioxide (CO_2) followed by air drying [15]. The aim is to assess these samples' performance and minimize water usage during manufacturing.

3.2.1. Tiles Cured/Dried in Air Alone

This set involves samples left to cure and dry naturally in the air.

Fig. 4 Air drying/curing of tiles

3.2.2. Sample Diffused with CO_2

This set of tiles is subjected to a new method where carbon dioxide is diffused into the samples, then dried and cured in the air. This method is being investigated to explore the potential benefits of CO_2 curing and reducing water usage.

3.3. Process of Diffusion of Co_2 into the Tiles

The steps for the CO_2 curing process of the prepared tiles inside the CO_2 curing chamber are as follows**:**

Place samples in the Chamber	Carefully place 6-8 samples inside the CO_2 curing chamber uniformly and expose them to the CO_2 gas.
Prevent Gas Leaks	Before initiating the CO_2 curing process, prevent any gas leaks. It is crucial to ensure the chamber is airtight to maintain the desired pressure during the curing process.
Increase Pressure to 2 kg/cm2	Gradually increase chamber pressure to 2 kg/cm2 by adjusting valves and CO_2 gas flow.
Regulate Pressure at 2 kg/cm2	Maintain a constant 2 kg/cm2 pressure by regulating gas flow. Monitor the pressure gauge regularly for consistency.
Check for Sudden Pressure Drops	Constantly monitor the pressure gauge for the 4-hour CO_2 curing process. Address sudden pressure drops promptly to rectify issues and maintain 2 kg/cm2 pressure.
Maintain Pressure for 4 Hours	Maintain 2 kg/cm2 pressure consistently for the full 4-hour CO_2 curing process to facilitate carbonation reaction and desired curing.

By following these steps and ensuring a controlled and stable CO_2 curing process, the samples can undergo the necessary carbonation to potentially enhance their properties and contribute to the reduction of environmental pollution through CO_2 sequestration [16]

Fig. 5 Carbon dioxide cylinder

Fig. 6 CO_2 curing chamber

3.3.1. Tiles Cured in Water and Air

These samples are cured using the traditional water immersion method for a specified duration.

3.3.2. CO_2 Curing Process

The samples were subjected to CO_2 diffusion for a period of 4 hours inside the CO_2 curing chamber.

3.3.3. Post-Curing Process

After the CO_2 diffusion, the tiles were removed from the chamber and cured in the air for 3 days and 7 days. This allows the tiles to develop their properties and strength over time after the CO_2 treatment.

3.3.4. Companion Tile Sets

Air-Cured Samples

Another set of tiles was cast alongside the CO_2-diffused tiles but not subjected to CO_2 treatment. Instead, they were cured in the air for 3 and 7 days. This set compares to the CO_2-cured tiles to assess any differences in performance.

Water-Cured Samples

A separate set of samples was cast, similar to the previous sets, but these samples were cured using the traditional method of water curing for 3 days and 7 days, following the existing methodology [17]. This allows for comparing the effects of CO_2 diffusion against traditional water curing. By investigating this manner, the study can effectively compare the efficacy of diffusing CO_2 into the tiles with other curing methods (air and water). It allows researchers to understand how CO_2 curing impacts the sample properties and strength compared to conventional curing approaches [15].

This comprehensive analysis will provide valuable insights into the potential benefits of CO_2 diffusion in tile manufacturing, contributing to both the development of sustainable practices and the reduction of environmental impact.

3.4. Testing for Transverse Strength

The samples exposed to carbon dioxide and curing were carried out in the air, and the companion samples cured in air and water were tested for transverse strength as per IS:1237-2012. The results of the tests are tabulated in Table 1.

Fig. 7 Testing of tiles for transverse strength in UTM

3.4. Qualitative Tests for Determination of Co_2 in the Tile

The presence of carbon dioxide (CO_2) in CO_2-cured samples can be confirmed through the Lime Water Test. This test involves exposing lime water to the surface of the CO_2-cured tile or the surrounding environment where the samples are stored [18]. The process is as follows:

3.4.1. Preparation of Lime Water

Lime water is a solution of calcium hydroxide ($Ca(OH)_2$) in water. It is prepared by dissolving a small amount of calcium hydroxide (lime) in water until it forms a clear solution.

3.4.2. Exposure to CO_2-Cured Tile

The lime water is then exposed to the CO_2-cured tile or the area around the tiles.

3.4.3. Observation of Color Change

When lime water comes into contact with carbon dioxide, it undergoes a chemical reaction to form calcium carbonate ($CaCO_3$), which is insoluble and appears as a white precipitate. This results in a visible color change of the lime water from colorless to white.

3.4.4. Confirmation of CO_2 Presence

The formation of a white precipitate confirms the presence of carbon dioxide in the CO_2-cured tile or the surrounding atmosphere. By sharing videos of the Lime Water Test through WhatsApp, researchers can visually demonstrate the confirmation of carbon dioxide presence in the CO_2-cured tiles [11] [19]. This qualitative test provides evidence of the carbonation process occurring in the tiles during the CO_2 curing process, which is crucial for understanding the effectiveness of the CO_2 curing method in the study.

Fig. 8 Lime water test

3.5. The Bromothymol Blue Indicator Test

It is another method to detect the presence of carbon dioxide (CO_2) in CO_2-cured tile powder. This test relies on the property of bromothymol blue, an indicator solution that changes color in the presence of CO_2. The procedure for the test is as follows:

3.5.1. Preparation of Bromothymol Blue Solution

Bromothymol blue is a pH indicator that is typically prepared as a solution in water. In its basic form, the solution appears blue.

3.5.2. Immersing CO2-Cured Tile Powder

The CO_2-cured tile powder is immersed in the bromothymol blue solution. As the powder reacts with the solution, any CO_2 present in the tile powder will influence the pH of the solution.

3.5.3. Color Change Observation

In the presence of CO_2, the bromothymol blue solution changes pH, causing it to shift from blue to a greenish or yellowish color. This color change indicates the presence of carbon dioxide in the CO_2-cured tile powder.

3.5.4. Confirmation of CO_2 Presence

The color change in the bromothymol blue solution confirms the carbon dioxide sequestered in the CO_2-cured sample powder. Using the Bromothymol Blue Indicator Test, researchers can visually demonstrate the presence of CO_2 in the CO_2-cured sample powder.

This qualitative test adds to the evidence of successful carbonation during the CO_2 curing process and provides valuable information about the efficacy of the CO_2 curing method in the study.

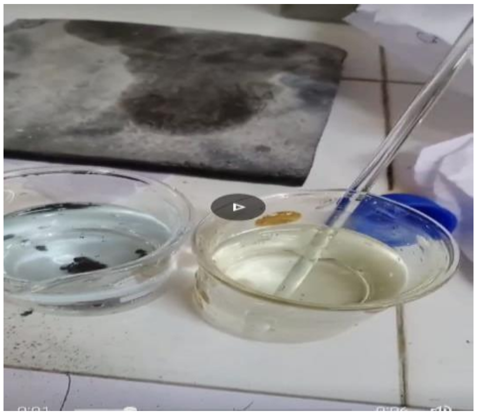


Fig. 9 The bromothymol blue indicator test

3.6. The Liquid Ammonia Test

This is another method used to detect the presence of carbon dioxide (CO_2) in CO_2-cured tile powder. This test relies on the reaction of CO_2 with liquid ammonia to form a white precipitate. The procedure for the test is as follows:

3.6.1. Preparation of Liquid Ammonia Solution

Liquid ammonia (ammonium hydroxide) is prepared as a solution in water. It is usually a colorless liquid with a strong ammonia odor.

3.6.2. Immersing CO_2-Cured Tile Powder

The CO_2-cured tile powder is immersed in the liquid ammonia solution.

3.6.3. Observation of White Precipitate

In the presence of CO_2, the ammonia solution reacts with the carbon dioxide to form ammonium carbonate ((NH4)2CO_3). Ammonium carbonate is an insoluble white solid, forming a white precipitate in the solution.

3.6.4. Confirmation of CO_2 Presence

The formation of a white precipitate confirms the presence of carbon dioxide in the CO_2-cured tile powder.

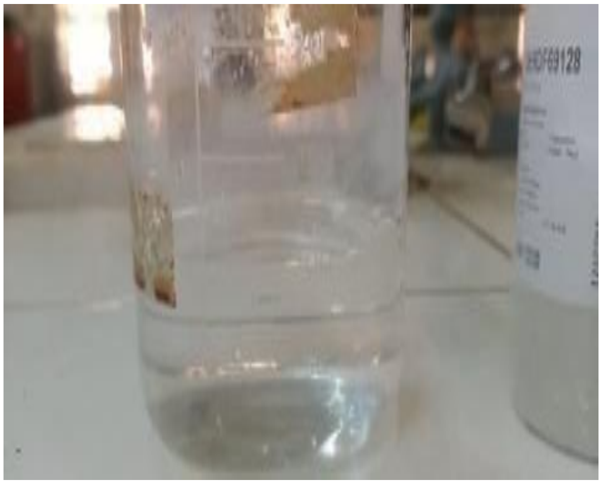


Fig. 10 The Liquid Ammonia Test

By using the Liquid Ammonia Test, researchers can visually demonstrate the presence of CO_2 in the CO2-cured tile powder. This qualitative test provides additional evidence of the carbonation process occurring in the tiles during the CO_2 curing process. Sharing videos of this test through WhatsApp allows others to witness and understand the test results, further supporting the study's efficacy of the CO_2 curing method.

3.7. The Phenolphthalein Indicator Test

This method is used to detect the presence of carbon dioxide (CO_2) in CO_2-cured sample powder [20]. This test relies on the pH sensitivity of phenolphthalein, a colorless indicator that undergoes a color change in the presence of CO_2. The procedure for the test is as follows:

3.7.1. Preparation of Phenolphthalein Solution

Phenolphthalein is prepared as a solution in water. In its basic form, the solution appears colorless.

3.7.2. Immersing CO_2-Cured Tile Powder

The CO_2-cured sample powder is immersed in the phenolphthalein solution.

3.7.3. Observation of Color Change

In the presence of CO_2, the phenolphthalein solution undergoes a change in pH, becoming slightly acidic. This change in pH causes the phenolphthalein to shift from colorless to pink or magenta.

3.7.4. Confirmation of CO_2 Presence

The color change to pink indicates the presence of carbon dioxide in the CO_2-cured sample powder.

By using the Phenolphthalein Indicator Test, researchers can visually demonstrate the presence of CO_2 in the CO_2-cured sample powder. This qualitative test provides additional evidence of successful carbonation during the CO_2 curing process.

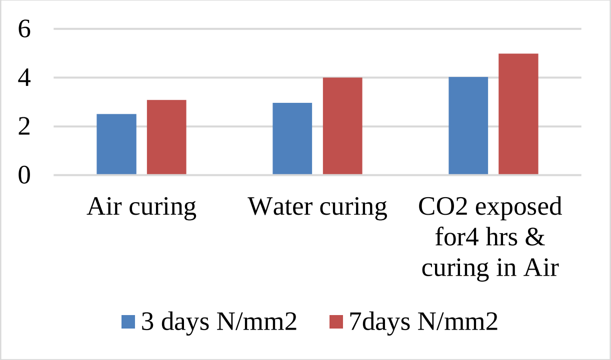
3.7.5. Indicator Test

Fig. 11 The phenolphthalein indicator test

3.8. Wet Transverse Strength

Table 1. Wet transverse strength of the sample

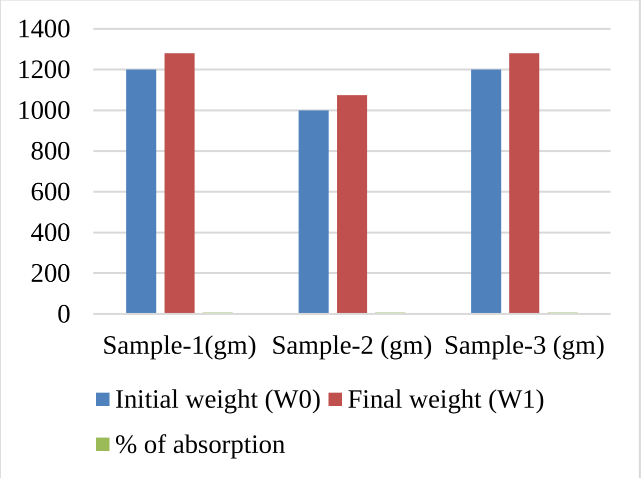
Different curings	3 days N/mm^2	7days N/mm^2
Air curing	2.5	3.08
Water curing	2.96	4.0
CO_2 exposed for 4 hrs and cured in Air	4.03	4.99



Graph 1. Showing the percentage of wet transverse strength of the sample

Table 2. The estimated carbon dioxide through BET analysis indicates that the amount of carbon dioxide absorbed as a percentage of CO2 absorption

	Sample-1 (gm)	Sample-2 (gm)	Sample-3 (gm)
Initial weight (W0)	1200	1000	1200
Final weight (W1)	1280	1075	1280
% of absorption	7	7.5	7



Graph 2. The graph shows the estimated carbon dioxide through BET analysis, indicating that the amount of carbon dioxide absorbed as a percentage of CO2 absorption

4. Discussion

From Tables 1 and 2 and Graphs 1 and 2, the study results show significant improvements in the early strength of the sample cured with CO_2 compared to the companion sample cured in air and water for both the 3 days and 7 days curing periods. The increase in strength is remarkable, with the CO_2-cured sample exhibiting more than a 50% increase in strength compared to the air-cured sample and more than a 30% increase compared to the water-cured sample. The charts visually represent the attained strength for the 3 days and 7 days curing periods. The data in these charts likely show the flexural strength values of the tiles for each curing method and curing duration. From the charts and results, it is evident that the sample diffused with CO_2 and cured in air performed exceptionally well, providing satisfactory results in terms of strength at both 3 days and 7 days. This performance surpasses that of their companion tiles cured in air and water. An essential aspect highlighted in the study is eliminating water usage in the CO_2 curing process. This indicates a significant advantage in sustainable tile manufacturing, as water needs during the curing stage are eliminated.

Overall, the findings suggest that the CO_2 curing process has proven to be highly effective in enhancing the early strength of the tiles while minimizing water usage and offering a promising approach for sustainable sample production. The visual representation through charts provides a clear and concise understanding of the strength improvements attained through CO_2 curing for 3-day and 7-day periods. Table 2 shows the average value of % absorption of CO_2 after curing the samples is about 7%. The estimated value of % absorption of CO_2 is about 0.08 kg/tile.

The qualitative tests conducted on the specimens have confirmed the presence of carbon dioxide (CO_2) in the tiles. The results of each test are as follows:

4.1. Lime Water Test

The color change from colorless to milky white in the lime water indicates the presence of CO_2 in the samples. This confirms that the CO_2 curing process led to carbonation in the samples.

4.2. Bromothymol Blue Indicator Test

The color change from yellow to blue in the bromothymol blue indicator test indicates the presence of CO_2 in the samples. This further confirms the successful carbonation of the samples during the CO_2 curing process.

4.3. Liquid Ammonia Test

The formation of a white precipitate in the liquid ammonia test indicates the presence of CO_2 in the samples. This provides additional evidence of carbon dioxide presence.

4.4. Phenolphthalein Indicator Test

The color change from colorless to pink in the phenolphthalein indicator test confirms the presence of CO_2 in the samples. This test further supports the successful carbonation of the samples during the CO_2 curing process.

The positive results obtained from these qualitative tests are consistent with the earlier findings and observations of increased strength in the CO_2-cured samples compared to companion samples cured in air and water. The successful confirmation of the presence of carbon dioxide through multiple qualitative tests provides strong evidence of the effectiveness of the CO_2 curing process in the study. It reinforces the understanding that the CO_2 curing method contributes to the development of improved sample properties and supports the reduction of environmental impact by sequestering CO_2 in the sample.

5. Conclusion

Based on the comprehensive tests and analyses conducted on the tiles, the following conclusions can be drawn:

- The CO_2-cured tiles exhibited significantly higher early strengths compared to companion tiles cured in air and water. The increase in strength was more than 50% compared to air curing and over 30% compared to water curing.
- Tiles cured with CO_2 and cured in air showed satisfactory results at both 3 days and 7 days compared to their companion tiles. Moreover, the CO_2 curing process eliminates the need for water, making it a more sustainable and water-efficient method.
- The qualitative analysis using multiple tests (lime water test, bromothymol blue indicator test, liquid ammonia test, and phenolphthalein indicator test) confirmed the successful sequestration of carbon dioxide in the CO_2-cured tiles.
- Quantitative analysis using both the BET analysis and weighing the sample before and after CO_2 diffusion indicated that approximately 0.8 kg of CO_2 was absorbed per tile. The results from both methods were consistent, validating the efficacy of CO_2 absorption.
- 5. The study's findings confirm that using carbon dioxide for tile curing improves early strength, reduces or eliminates the need for water in the curing process, and contributes to reducing carbon emissions during sample manufacturing.

In summary, using CO_2 in the curing process of samples has shown promising results in terms of strength enhancement, water conservation, and carbon dioxide sequestration. These findings suggest that CO_2 curing can be a sustainable and eco-friendly alternative for tile manufacturing, contributing to environmental preservation and improved manufacturing practices.

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