

Experimental Study on Carbonation of Concrete with Different Dimensional CO₂ Ingress

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Abstract: Concrete alkalinity protects the reinforcement bars from corrosion but the carbonation phenomenon significantly contributes to the destruction of their passive coating, thus favouring the corrosion onset. Carbonation of concrete decreases the pH value of the concrete which can initiate the corrosion of reinforcements. Reinforcement corrosion is mainly responsible for reinforced concrete degradation. The parameters like dimensional carbon dioxide ingress, duration of carbon dioxide ingress, grade of concrete and volumetric concentration of CO₂ are considered for this thesis work. After 28 days curing, specimens were placed in the chamber having supply of carbon dioxide gas. The specimens after exposed in carbon dioxide chamber for two different durations are 145 hours and 290 hours. Then the specimens were cut at 1cm, 2cm and 3 cm respectively from the concrete surface. Those concrete specimens were crushed, powdered, sieved by 90µm sieve and the powder which passed through the 90µm sieve was collected as sample powder. Carbonation depth has been analysed using phenolphthalein indicator and Fourier Transform Infrared Technique (FT-IR). The measurement of carbonation depth using Infrared spectroscopy was done by referring the characteristic peak of the C-O bonds from the sample powder. The measurement of carbonation depth using the phenolphthalein solution was carried out by spraying the indicator on the split surface of the concrete specimens

Keywords: Carbonation, carbon dioxide ingress, phenolphthalein indicator, and carbonation depth

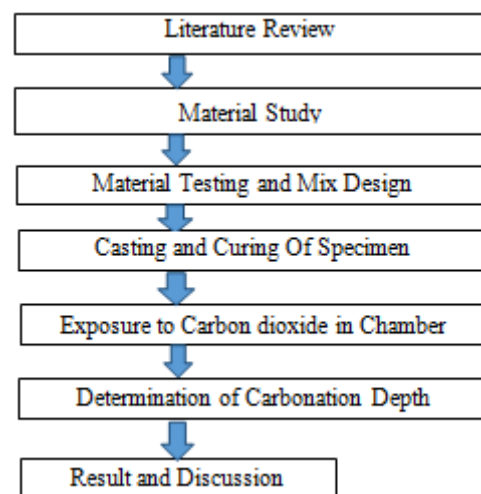
1. Introduction

1.1 Carbonation

Reinforced structures are subject to deterioration over time. The degradation of their mechanical characteristics jeopardize their functional capacity. This degradation may occur due to in-service conditions, exposure to aggressive environmental agents, and inadequate use or maintenance condition. The concrete degradation mechanisms can be physical, chemical or mechanical, and the chemical and physical phenomena may be synergetic. The cause of deterioration of reinforced concrete that deserves most attention is reinforcement corrosion. It is one of the most important pathological manifestations to affect reinforced concrete structures, and is difficult to intervene or repair. The alkalinity of concrete protects the reinforcement from corrosion until chemical or physical changes occur that enable external aggressive agents to act. The two main agents initiate reinforcement corrosion by destroying its passive coating: carbonation and chloride ingress. The concrete carbonation is one of the main phenomena to initiate the process of reinforcement corrosion. Concrete carbonation begins with the penetration of atmospheric CO₂(g) across its surface and its subsequent diffusion inside the material, where it dissolves in the aqueous phase (CO₂(aq)). The result of solubilisation is the formation of ionic species (HCO₃⁻ and especially CO₃²⁻) that interact with the various calcium-bearing phases of the cement, primarily portlandite, leading to the precipitation of calcium carbonate, normally in the form of calcite. Carbonation is not in itself harmful and in some cases may even be beneficial, for it reduces porosity and forms a protective layer on the concrete surface. Carbonation is characterized by a physical-chemical process in which a series of chemical reactions occur in the presence of carbon dioxide (CO₂), which fosters the reduction of pH in concrete. CO₂ penetrates concrete

predominantly through a diffusion mechanism. This penetration and carbonation reaction occurs gradually, leading to a carbonated layer (limited by the so-called carbonation front) that increases in thickness over time. It is generally agreed that carbonation does not occur in the same way in all mixes, nor does it occur in all circumstances; different mixes will exhibit distinct carbonation and the same mix exposed to different environments will not show the same carbonation. Reinforced concrete carbonates from the surface inward, advancing across the pores until the carbonation front reaches the steel. In all the many models described in the literature for predicting carbonation depth, CO₂(g) ingress depends on the porosity of the material and its water content. A priori, higher concrete porosity may be thought to induce speedier carbonation front progress and greater loss of alkalinity (porous concretes carbonate very rapidly).

2. Methodology



Volume 5 Issue 11, November 2016

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2.1 Mix Design

In this thesis work, mix design has been done using Indian Standard Recommended method of Concrete Mix Design (IS 10262-2009)

M20 – 1:2.26:3.95 at 0.45 w/c ratio

M25 – 1:2.03:3.33 at 0.4 w/c ratio

2.2 Casting of Specimen

Size of cylinder is 150mm x 300mm. Initially the moulds were cleaned and oiled. As per designed proportions all the ingredients such as cement and fine aggregate, coarse aggregate and water were weighed. Initially cements and fine aggregate were thoroughly mixture was thoroughly mixed and then coarse aggregate was added. Then the mixture was thoroughly mixed until the coarse aggregate is uniformly distributed. Finally the water was added. Now the mould was placed on the vibrator and compacted well. After the surface was finished and the specimens were numbered. Then the test specimens were numbered. Then the test specimens were placed in room temperature for 24 hours. After 24 hours the specimen were demoulded and allowed to cure for 28 days. The below table shows the number of cubes and cylinder.

Table 1: Casting of specimen

Grade	Number of cylinder		Number of cubes		Control specimen
	1D	2D	3D (145 hours)	3D (290 hours)	
M20	6	6	3	3	6
M25	6	6	3	3	6

2.3 Carbonation Chamber

Carbonation chamber is made of acrylic sheet. It is easy available in local market. Size of the chamber is 6 ft x 4 ft x 2 ft. Carbon dioxide is passed into the chamber, the carbon dioxide was circulated uniformly throughout the chamber and the effect of carbonation will be uniform. The carbon dioxide cylinder is attached with heater to provide good flow of CO₂ into the chamber without formation of ice in the cylinder.

2.4 Duration of Carbon Dioxide Purging:

The specimens were placed inside the carbonation chamber in presence of carbon dioxide for the certain time period. Carbonation was started from 15/02/16 to 21/04/16. The total carbonation time period is 290.15 hours. In the carbonation time period the specimens were kept in the carbonation chamber.

3. Experimental Work

3.1 Determination of Carbonation Depth

We have adopted two techniques to determination of carbonation depth they are

- Phenolphthalein indicator
- FT-IR Spectroscopy

The traditional way of determine the depth of carbonation use to spray phenolphthalein indicator on to the surface of

concrete. FR-IR has also been too used to detect the spectral changes associated with the presence of Ca(OH)₂ at the interface between the cement paste and aggregate.

3.2 Phenolphthalein Indicator

Carbonation depth is assessed using a solution of phenolphthalein indicator that appears pink in contact with alkaline concrete with pH values in excess of 9 and colorless at lower levels of pH. There are three tests in the phenolphthalein indicator testing they are

- Splitting of Cube and cylinder.
- Preparation of phenolphthalein indicator.
- Spraying of phenolphthalein in splitted cubes and cylinders.

3.2.1 Splitting of Cubes and Cylinders

The cylinders and the cubes were taken out from the chamber after 60 days. The cylinders were split in tensile splitting test and the cubes where split in compression testing machine.

3.2.2 Preparation of Phenolphthalein Indicator

The indicator which is used to find the carbonation depth is phenolphthalein indicator. The procedure for preparation of phenolphthalein indicator is to mix the ethanol solution (50%) and distilled water (49%) with 1% of phenolphthalein powder.

3.2.3 Spraying of Phenolphthalein in Splitted Cubes and Cylinders

The transverse exposed surface were immediately cleaned and then sprayed with the phenolphthalein indicator. In the noncarbonated part of the specimen, where the concrete was still highly alkaline, a purple-red color was obtained. In the carbonated part of the specimen where the alkalinity of concrete is reduced, no coloration occurred. The selected specimens were marked by pen at 1cm, 2cm, and 3cm respectively from the outer surface. Then the specimen were cut at 1cm, 2cm, 3cm from the outer surface and power well. From the powdered sample, carbonation depth has been measures using FT-IR spectroscopy.

3.3 FT-IR Spectroscopy

FR-IR spectroscopy is a powerful tool for determining the structure of the functional groups that build up the molecules. From the FT-IR Spectroscopy results the characteristic peak of the C-O functional group was identify. From the C-O bond results the carbonation in a complex concrete composite was determined.

4. Results and Discussion

4.1 Analysis of Carbonation Depth Using Phenolphthalein Indicator

4.1.1 Analysis of Average Carbonation Depth Using Phenolphthalein Indicator:

Table 2: Analysis of Average Carbonation Depth Using Phenolphthalein Indicator

Grade	Carbonation depth	Average carbonation depth(cm)
Control specimen M ₂₀	0.4,0.5,0.6	0.5
M20 1D	2.5, 2, 3	2.5
M20 2D	2.8, 2.6, 2.5	2.7
M20 3D	2.7, 3, 3	2.9
Control specimen M ₂₅	0.3,0.4,0.5	0.4
M25 1D	2, 1.75, 1.9	1.9
M25 2D	2.4, 2.1, 1.75	2.1
M25 3D	3, 2, 3	2.6

In M20 grade (3D) specimen the carbonation depth was increased by 5 times when compared to control specimen M20. In M25 grade (3D) specimen the carbonation depth was increased by 4.75 times when compared to control specimen 25. In M20 grade (3D) specimen the carbonation depth was increased by 1.16 times when compared to M20 grade 1D. In M25 grade (3D) specimen the carbonation depth was increased by 1.36 times when compared to M25 grade 1D. In M20 grade (2D) specimen the carbonation depth was increased by 1.08 times when compared to M20 grade 1D. In M25 grade (2D) specimen the carbonation depth was increased by 1.105 times when compared to M25 grade 1D. Carbonation depth increases with water cement ratio and the age of the concrete. From the results, it is clear that grade of concrete acts as a vital parameter during the carbonation of concrete. Grade of concrete more than M20 performs better when compared with M20.

4.1.2 Analysis of Maximum Carbonation Depth Using Phenolphthalein Indicator:

Table 3: Analysis of Maximum Carbonation Depth Using Phenolphthalein Indicator

Grade	Maximum Carbonation depth(cm)
Control specimen M ₂₀	0.7
M20 1D	2.5
M20 2D	2.8
M20 3D	3
Control specimen M ₂₅	0.5
M25 1D	2
M25 2D	2.4
M25 3D	3

In M20 grade (3D) specimen the carbonation depth was increased by 3.57 times when compared to control specimen M20. In M25 grade (3D) specimen the carbonation depth was increased by 4 times when compared to control specimen 25. In M20 grade (3D) specimen the carbonation depth was increased by 1.2 times when compared to M20 grade (1D) specimen. In M25 grade (3D) specimen the carbonation depth was increased by 1.5 times when compared to M25 grade (1D) specimen. In M20 grade (2D) specimen the carbonation depth was increased by 1.12 times when compared to M20 grade (1D) specimen. In M25 grade (2D) specimen the carbonation depth was increased by 1.2 times when compared to M25 grade (1D) specimen. The measurements of carbonation depth using the phenolphthalein solution was carried out by spraying the indicator on the split surface of the cube and cylinder. The phenolphthalein color indicator only detects pH value. In the noncarbonated part of the specimen, where the concrete was

still highly alkaline, a purple-red color was obtained. In the carbonated part of the specimen where the alkalinity of the concrete is reduced, no coloration occurred. Carbonation depth increases with water cement ratio and the age of the concrete. From the results, it is clear that grade of concrete acts as a vital parameter during the carbonation of concrete. Grade of concrete more than M20 performs better when compared with M20.

4.2 Fourier Transform Infrared Spectroscopy Result:

4.2.1 FT-IR graph for M20 grade of concrete (1D)

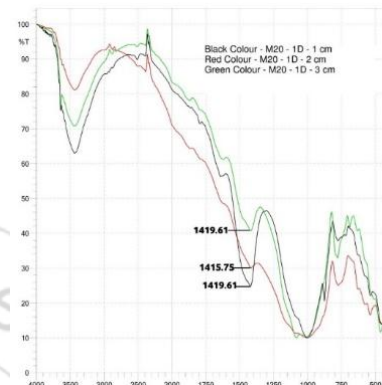


Figure 1: FT-IR graph for M20 grade of concrete (1D)

From this figure Carbonation depth is determined from the position of the C-O characteristic peaks comparative to the baseline at wave number is 1419.61 cm⁻¹ at 1cm depth. The percentage of higher transmittance at a wave length 1415.75 cm⁻¹ at 2cm depth of the cylinder which represents the C-O bonding. The percentage of higher transmittance at a wave length 1419.61 cm⁻¹ at 3cm depth of the cylinder which represents the C-O bonding.



Figure 2: FT-IR graph for M20 grade of concrete (1D)

From this figure Carbonation depth is determined from the position of the C-O characteristic peaks comparative to the baseline at wave number is 1440.25 cm⁻¹ at 1cm depth. The percentage of higher transmittance at a wave length 1483.26 cm⁻¹ at 2cm depth of the cylinder which represents the C-O bonding. The percentage of higher transmittance at a wave length 1421.54 cm⁻¹ at 3cm depth of the cylinder which represents the C-O bonding.

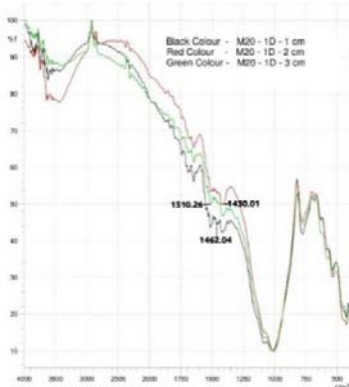


Figure 3: FT-IR graph for M20 grade of concrete (1D)

From this figure Carbonation depth is determined from the position of the C-O characteristic peaks comparative to the baseline at wave number is 1462.04 cm^{-1} at 1cm depth. The percentage of higher transmittance at a wave length 1430.01 cm^{-1} at 2cm depth of the cylinder which represents the C-O bonding. The percentage of higher transmittance at a wave length 1510.26 cm^{-1} at 3cm depth of the cylinder which represents the C-O bonding.

4.2.2 FT-IR graph for M20 grade of concrete (2D):

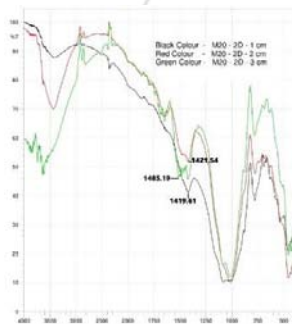


Figure 4: FT-IR graph for M20 grade of concrete (2D)

From this figure Carbonation depth is determined from the position of the C-O characteristic peaks comparative to the baseline at wave number is 1419.61 cm^{-1} at 1cm depth. The percentage of higher transmittance at a wave length 1421.54 cm^{-1} at 2cm depth of the cylinder which represents the C-O bonding. The percentage of higher transmittance at a wave length 1485.19 cm^{-1} at 3cm depth of the cylinder which represents the C-O bonding.

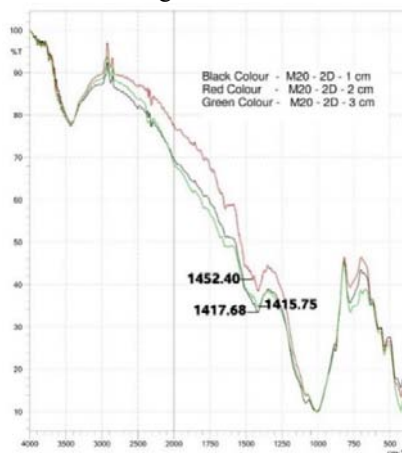


Figure 5: FT-IR graph for M20 grade of concrete (2D)

From this figure Carbonation depth is determined from the position of the C-O characteristic peaks comparative to the baseline at wave number is 1417.68 cm^{-1} at 1cm depth. The percentage of higher transmittance at a wave length 1452.40 cm^{-1} at 2cm depth of the cylinder which represents the C-O bonding. The percentage of higher transmittance at a wave length 1415.75 cm^{-1} at 3cm depth of the cylinder which represents the C-O bonding.

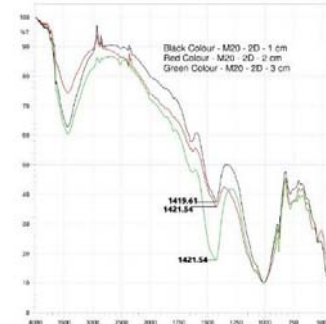


Figure 6: FT-IR graph for M₂₀ grade of concrete (2D)

From this figure Carbonation depth is determined from the position of the C-O characteristic peaks comparative to the baseline at wave number is 1419.61 cm^{-1} at 1cm depth. The percentage of higher transmittance at a wave length 1421.54 cm^{-1} at 2cm depth of the cylinder which represents the C-O bonding. The percentage of higher transmittance at a wave length 1421.54 cm^{-1} at 3cm depth of the cylinder which represents the C-O bonding.

4.2.3 FT-IR graph for 145 hours carbonated M20 grade of concrete (3D) specimen:

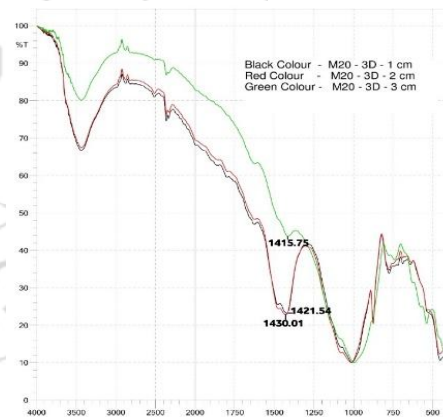


Figure 7: FT-IR graph for 145 hours carbonated M20 grade of concrete (3D) specimen

From this figure Carbonation depth is determined from the position of the C-O characteristic peaks comparative to the baseline at wave number is 1421.54 cm^{-1} at 1cm depth. The percentage of higher transmittance at a wave length 1430.01 cm^{-1} at 2cm depth of the cube which represents the C-O bonding. The percentage of higher transmittance at a wave length 1415.75 cm^{-1} at 3cm depth of the cube which represents the C-O bonding.

4.2.4 FT-IR graph for 290 hours carbonated M20 grade of concrete (3D) specimen:

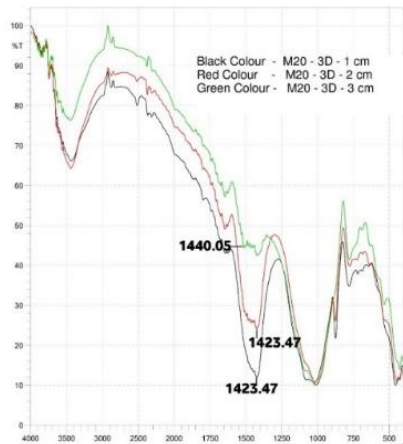


Figure 8: FT-IR graph for 290 hours carbonated M20 grade of concrete (3D) specimen

From this figure Carbonation depth is determined from the position of the C-O characteristic peaks comparative to the baseline at wave number is 1423.47 cm^{-1} at 1cm depth. The percentage of higher transmittance at a wave length 1423.47 cm^{-1} at 2cm depth of the cube which represents the C-O bonding. The percentage of higher transmittance at a wave length 1440.05 cm^{-1} at 3cm depth of the cube which represents the C-O bonding.

4.2.5 FT-IR graph for M25 grade of concrete (1D):

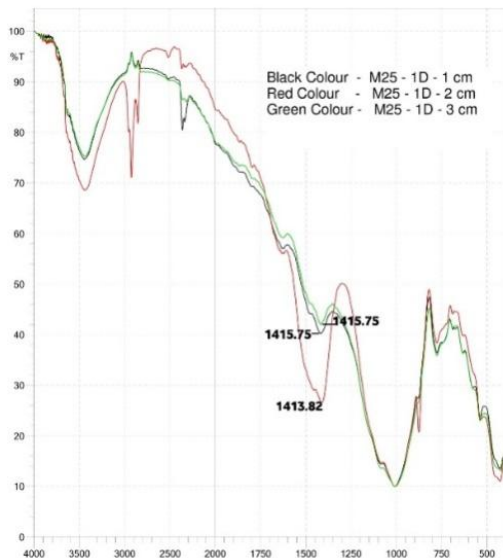


Figure 9: FT-IR graph for M25 grade of concrete (1D)

From this figure Carbonation depth is determined from the position of the C-O characteristic peaks comparative to the baseline at wave number is 1415.75 cm^{-1} at 1cm depth. The percentage of higher transmittance at a wave length 1413.82 cm^{-1} at 2cm depth of the cylinder which represents the C-O bonding. The percentage of higher transmittance at a wave length 1415.75 cm^{-1} at 3cm depth of the cylinder which represents the C-O bonding.

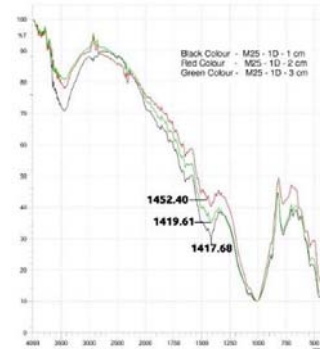


Figure 10: FT-IR graph for M25 grade of concrete (1D)

From this figure Carbonation depth is determined from the position of the C-O characteristic peaks comparative to the baseline at wave number is 1417.68 cm^{-1} at 1cm depth. The percentage of higher transmittance at a wave length 1452.40 cm^{-1} at 2cm depth of the cylinder which represents the C-O bonding. The percentage of higher transmittance at a wave length 1419.61 cm^{-1} at 3cm depth of the cylinder which represents the C-O bonding.



Figure 11: FT-IR graph for M25 grade of concrete (1D)

From this figure Carbonation depth is determined from the position of the C-O characteristic peaks comparative to the baseline at wave number is 1417.68 cm^{-1} at 1cm depth. The percentage of higher transmittance at a wave length 1419.61 cm^{-1} at 2cm depth of the cylinder which represents the C-O bonding. The percentage of higher transmittance at a wave length 1419.61 cm^{-1} at 3cm depth of the cylinder which represents the C-O bonding.

4.2.6 FT-IR graph for M25 grade of concrete (2D):

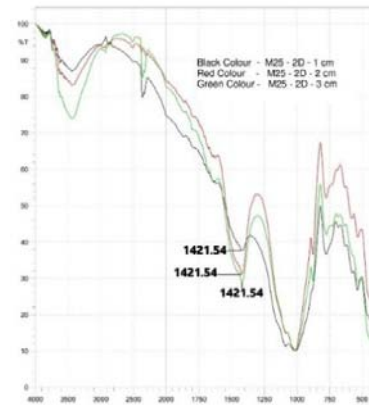


Figure 12: FT-IR graph for M25 grade of concrete (2D)

From this figure Carbonation depth is determined from the position of the C-O characteristic peaks comparative to the baseline at wave number is 1421.54 cm^{-1} at 1cm depth. The percentage of higher transmittance at a wave length 1421.54 cm^{-1} at 2cm depth of the cylinder which represents the C-O bonding. The percentage of higher transmittance at a wave length 1421.54 cm^{-1} at 3cm depth of the cylinder which represents the C-O bonding.

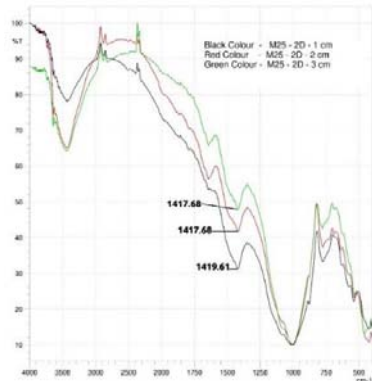


Figure13: FT-IR graph for M₂₅ grade of concrete (2D)

From this figure Carbonation depth is determined from the position of the C-O characteristic peaks comparative to the baseline at wave number is 1419.61 cm^{-1} at 1cm depth. The percentage of higher transmittance at a wave length 1417.68 cm^{-1} at 2cm depth of the cylinder which represents the C-O bonding. The percentage of higher transmittance at a wave length 1417.68 cm^{-1} at 3cm depth of the cylinder which represents the C-O bonding.



Figure14: FT-IR graph for M₂₅ grade of concrete (2D)

From this figure Carbonation depth is determined from the position of the C-O characteristic peaks comparative to the baseline at wave number is 1417.68 cm^{-1} at 1cm depth. The percentage of higher transmittance at a wave length 1413.82 cm^{-1} at 2cm depth of the cylinder which represents the C-O bonding. The percentage of higher transmittance at a wave length 1415.75 cm^{-1} at 3cm depth of the cylinder which represents the C-O bonding.

4.2.7 FT-IR graph for 145 hours carbonated M25 grade of concrete (3D) specimen:



Figure 15: FT-IR graph for 145 hours carbonated M25 grade of concrete (3D) specimen

From this figure Carbonation depth is determined from the position of the C-O characteristic peaks comparative to the baseline at wave number is 1406.11 cm^{-1} at 1cm depth. The percentage of higher transmittance at a wave length 1415.75 cm^{-1} at 2cm depth of the cube which represents the C-O bonding. The percentage of higher transmittance at a wave length 1409.96 cm^{-1} at 3cm depth of the cube which represents the C-O bonding.

4.2.8 FT-IR graph for 290 hours carbonated M25 grade of concrete (3D) specimen:

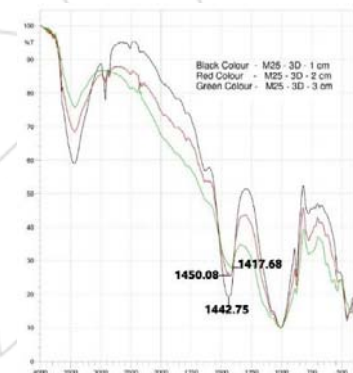


Figure16: FT-IR graph for 290 hours carbonated M25 grade of concrete (3D) specimen

From this figure Carbonation depth is determined from the position of the C-O characteristic peaks comparative to the baseline at wave number is 1442.75 cm^{-1} at 1cm depth. The percentage of higher transmittance at a wave length 1450.08 cm^{-1} at 2cm depth of the cube which represents the C-O bonding. The percentage of higher transmittance at a wave length 1417.68 cm^{-1} at 3cm depth of the cube which represents the C-O bonding

5. Conclusion

Increase in atmospheric carbon dioxide emissions will increase the rate of carbonation in reinforced concrete structures. In general, carbon dioxide present in the atmosphere permeates into concrete and carbonates the concrete and reduces the alkalinity of concrete. As the pH value of the concrete decreases, the protective layer gets destroyed and the steel begins to corrode. Hence in thesis work, we have studied the factors influencing of the carbonation in concrete. The following conclusions can be

drawn from this investigation. In M20 grade (3D) specimen the carbonation depth was increased by 3.57 times when compared to control specimen M20. In M25 grade (3D) specimen the carbonation depth was increased by 4 times when compared to control specimen 25.

- In M₂₀ grade 3D specimen the average carbonation depth increases 1.16 times when compared to M₂₀ grade 1D specimen.
- In M₂₅ grade 3D specimen the average carbonation depth increases 1.36 times when compared to M₂₅ grade 1D specimen.
- In M₂₀ grade 2D specimen the average carbonation depth increases 1.08 times when compared to M₂₀ grade 1D specimen.
- In M₂₅ grade 2D specimen the average carbonation depth increases 1.105 times when compared to M₂₅ grade 1D specimen.
- In M₂₀ grade 3D specimen the maximum carbonation depth increases 1.2 times when compared to M₂₀ grade 1D specimen.
- In M₂₅ grade 3D specimen the maximum carbonation depth increases 1.5 times when compared to M₂₅ grade 1D specimen.
- M₂₀ grade 2D specimen the maximum carbonation depth increases 1.12 times when compared to M₂₀ grade 1D specimen.
- M₂₅ grade 2D specimen the maximum carbonation depth increases 1.2 times when compared to M₂₅ grade 1D specimen.
- From the results, it is clear that grade of concrete acts as a vital parameter during the carbonation of concrete. Grade of concrete more than M₂₅ perform better when compared with M₂₀.
- Carbonation depth increases with increase in duration of carbon dioxide exposure.
- Concrete was powdered and collected from M₂₀ and M₂₅ grade of concrete specimens. The samples are collected from the cubes and cylinders at various depths like 1cm, 2cm, and 3cm and are tested for FT-IR.
- From the FTIR results the M₂₀ grade concrete has got reasonably greater carbonation depth compared to M₂₅.
- From the results, it is observed that M₂₀ grade 1D specimen is having higher transmittance rate at 1cm than the M₂₀ grade 1D specimen of concrete at 2cm and 3cm.
- From the results, it is observed that M₂₀ grade 2D specimen is having higher transmittance rate at 3cm than the M₂₀ 2D specimen grade of concrete at 1cm and 2cm.
- From the results, it is observed that M₂₀ grade 3D specimen is having higher transmittance rate at 3cm than the M₂₀ 3D specimen grade of concrete at 1cm and 2cm.
- From the results, it is observed that M₂₅ grade 1D specimen is having higher transmittance rate at 2cm than the M₂₅ grade 1D specimen of concrete at 1cm and 3cm.
- From the results, it is observed that M₂₅ grade 2D specimen is having higher transmittance rate at 1cm than the M₂₅ grade 2D specimen of concrete at 2cm and 3cm.
- From the results, it is observed that M₂₅ grade 3D specimen is having higher transmittance rate at 2cm than the M₂₅ grade 3D specimen of concrete at 1cm and 3cm.

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