

Experimental study on carbonation depths of OPC and PPC concrete by conventional and advanced methods

Alok Ranjan^a, Shashi Kumar^{b*}, Rajesh Kumar^c & Devendra Mohan^c

^aDepartment of Civil Engineering, Bharat Heavy Electricals Limited, Barh 803 215 India

^bDepartment of Civil Engineering, Sitamarhi Institute of Technology, Sitamarhi 843 302 India

^cDepartment of Civil Engineering, Indian Institute of Technology (BHU), Varanasi 221005 India

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Analysis of carbonation depths in concrete through conventional phenolphthalein indicator method as well as advanced methodologies such as X-ray diffraction analysis (XRDA) and fourier transform infrared (FTIR) method has been used to evaluate the accurate effect of carbonation on concrete samples. Concrete is carbonated in an acceleration chamber for the experimental process to achieve results in a limited period. Concrete samples have been prepared by using OPC cement and PPC cement at three different water/cement ratios (0.40, 0.45, and 0.50), and retained in a carbonation chamber at fixed parameters. The results of carbonation depths of concrete samples have been obtained from advanced methods as well as conventional phenolphthalein indicator method and analyzed the results. Carbonation depths obtained from advanced methods have been nearly double that of results obtained from the conventional method for OPC and PPC concrete. The carbonation depth of concrete samples is increased with an increase in the W/C ratio. The carbonation depth of PPC concrete samples is higher in comparison to OPC concrete obtained by all mentioned three methods. Hence OPC concrete is proposed to use in place of PPC concrete to reduce the risk of carbonation in reinforced concrete structures in the polluted area.

Keywords: Concrete, Durability, Reinforcement corrosion, Carbonation, Phenolphthalein, X-Ray Diffraction Analysis, Fourier Transform Infrared

1 Introduction

Concrete is the most extensively used for infrastructure construction. The durability of concrete structures has a major problem due to the environmental impact on the concrete infrastructure. The durability of concrete depends on multiple factors¹. Among the most pressing concerns for structural concrete, durability is the corrosion of reinforcement² which may be caused by carbonation of concrete³ exposed to natural atmospheric conditions [Carbon dioxide (CO₂), humidity, temperature]. In moist conditions, the pH of the liquid of the pores in the concrete decreases due to a reaction between atmospheric carbon dioxide and the presence of alkaline ingredients in the concrete cement. Therefore, embedded reinforcement is corroding in these conditions. In the surroundings of the polluted area, the deterioration of concrete structures is a major problem due to the carbonation of the concrete. It is a very challenging task to the exact evaluation of effect of carbonation in concrete structures. The natural process of carbonation of

concrete structures in the surroundings is very slow while influences of accelerated carbonation on the concrete simulate the accurate performance of the effect of carbonation on the concrete⁴.

The phenolphthalein indicator method is the most cost-effective, fast, and conventional methodology for the evaluation of carbonation depth in concrete. However, the actual results of carbonation depth are not evaluated by this method. Old suggestions have revealed that CO₂ could react at depths more than those specified by a phenolphthalein indicator. While most researchers use phenolphthalein indicators to evaluate the carbonation depth, other advanced methods of the experiment such as XRDA and Fourier Transform Infra-Red FTIR method have been successfully used by some researchers. Lo and Lee⁵ studied the rate of carbonation on the concrete, and found that results obtained by Infrared spectrum analysis were 23.9 % more in comparison to results obtained by phenolphthalein indicator. Chang and Chen⁶ concluded that the carbonation front from XRDA methods was approximately double that found by the phenolphthalein indicator method. W/C ratio has a conspicuous effect on concrete

*Corresponding author (E-mail: shashi351@gmail.com)

Table 1 — Prepared mix proportion of OPC concrete samples

Mix	W/C	W (Kg /m ³)	C (Kg /m ³)	FA (Kg /m ³)	CA ₁₀ (Kg /m ³)	CA ₂₀ (Kg /m ³)	Total wt (Kg)
O ₁	0.5	197	394	660.03	451.96	677.95	2380.94
O ₂	0.45	197	438	629.08	449.55	674.33	2387.74
O ₃	0.4	197	493	595.57	444.37	666.56	2396.00

Table 2 — Prepared mix proportion of PPC concrete samples

Mix	W/C	W (Kg /m ³)	C (Kg /m ³)	FA (Kg /m ³)	CA ₁₀ (Kg /m ³)	CA ₂₀ (Kg /m ³)	Total wt (Kg)
P ₁	0.5	197	394	662	436	645	2334
P ₂	0.45	197	438	715	391	595	2336
P ₃	0.4	197	493	601	417	651	2359

carbonation irrespective of the mix design of concrete^{7,8}. An accelerated carbonation test was conducted at five different CO₂ concentration levels of 2% to 100% by volume, at a fixed temperature of 20°C and RH of 70%⁹. The rate of carbonation was found with a variation of fly ash and silica fume in concrete mixes at variable W/C ratios of 0.4 to 0.6¹⁰. Zhao et al.¹¹ studied the influence of longer periods of curing on the performance of carbonation resistance of concrete with fly ash and found longer periods of curing of concrete decrease the carbonation depth.

This article deals carbonation of Ordinary Portland Cement (OPC) and Portland Pozzolana Cement (PPC) concrete samples with variable W/C ratios by both conventional phenolphthalein indicator methods as well as XRDA and FTIR methods. Concrete samples are prepared at three different W/C ratios 0.4, 0.45, and 0.5. An accelerated carbonation chamber was used for the progress of carbonation of concrete samples under controlled environmental conditions of 4 % CO₂ concentration, 65 % humidity, 25 +/- 5 degree Celsius temperature, and 1atm pressure. The paper also presents the comparative study of carbonation depth determined by conventional and advanced methods with variable W/C ratios for OPC and PPC concrete.

2 Materials and Methods

2.1 Materials

In this study, OPC conforming to Indian Standard IS: 8112-1989¹² has been used in this research work. Aggregates conforming to IS: 383-1970¹³ consisting of the coarse aggregate of 10 mm and 20 mm nominal size and fine aggregate (sand) have been used. OPC concrete samples were prepared with the following mix proportion in line with IS: 456-2000¹⁴ and IS: 10262-2009¹⁵ as shown in Table 1. PPC concrete samples were prepared with the following mix proportion as shown in Table 2.

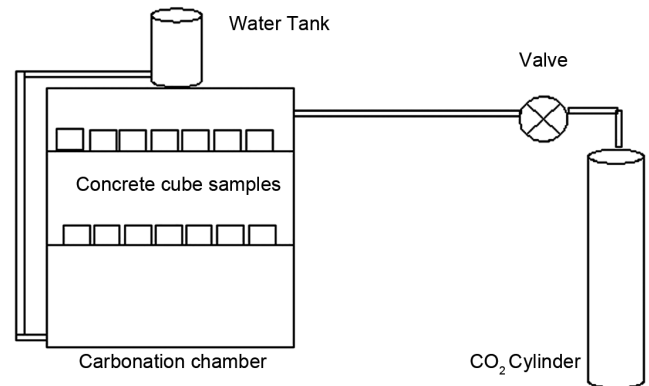


Fig. 1 — Schematic diagram of carbonation chamber.

2.2 Set-up for carbonation of concrete

An accelerated carbonation system^{16,17} has been used for the carbonation of concrete in a limited time. The carbonation chamber was used to retain concrete samples in a controlled environment. A constant concentration of 4 % CO₂, 65 % humidity, 25 +/- 5 degree Celsius temperature, and 1 atm pressure was maintained in the chamber during the experiment as shown in Fig. 1. CO₂ is constantly supplied to the chamber from a CO₂ cylinder. Water is kept in a container over the apparatus, and is connected to the same through an inlet pipe to maintain desired humidity inside the chamber. Concrete cubes cured for 3 days were placed in a carbonation chamber for 20 days. On completion of carbonation in the chamber, the cubes were drawn off from the chamber and sliced into pieces and tested for carbonation by the Phenolphthalein indicator method, XRDA, and FTIR methods.

2.3 Methodology

2.3.1 Phenolphthalein indicator method

The pH range of OPC concrete is 8.2 to 10, wherein the Phenolphthalein indicator remains colorless. The pH of the concrete medium is in the range of 12 to 13, wherein the color of the Phenolphthalein indicator is pink. Carbonation

involves the consumption of $\text{Ca}(\text{OH})_2$ in concrete resulting in the decrease of pH below 9.5 when the indicator turns colorless. In this experiment, the sliced parts of the carbonated samples were cleaned, and the phenolphthalein pH indicator was sprayed on their surface. Carbonated parts were identified to be colorless whereas the non-carbonated parts of the samples turned pink on spraying the indicator. Carbonation depth was averaged for taken multiple readings after five minutes of spraying of colorless zones (A1, A2, A3, A4, A5, A6, A7 & A8) as shown in Fig. 2 from the edge of concrete samples.

2.3.2 XRDA method

The X-ray diffraction analysis method is used for recognizing the atomic and molecular structure of a crystal. A cathode ray tube is used to throw Monochromatic X-rays on a crystalline sample resulting in constructive interference to produce a diffraction pattern as per Bragg's law ($n\lambda = 2d \sin \theta$), where n , λ , d and θ are respectively positive integer, a wavelength of the incident wave, scattering and inter-planar distance. The XRDA was conducted on model Mini Flex 600 as shown in Fig. 3. An operating condition with radiation (40 kV, 15 mA) and scan rate: 10 degree/min; 2θ : 10–90 degree was maintained. The peaks for CaCO_3 and $\text{Ca}(\text{OH})_2$ are used to find the extent of carbonation in concrete. For example, the existence of a peak for CaCO_3 exists, and no peak for $\text{Ca}(\text{OH})_2$ indicates that the sample is fully carbonated, while a peak for $\text{Ca}(\text{OH})_2$ and no peak for CaCO_3 indicates that the specimen is non-

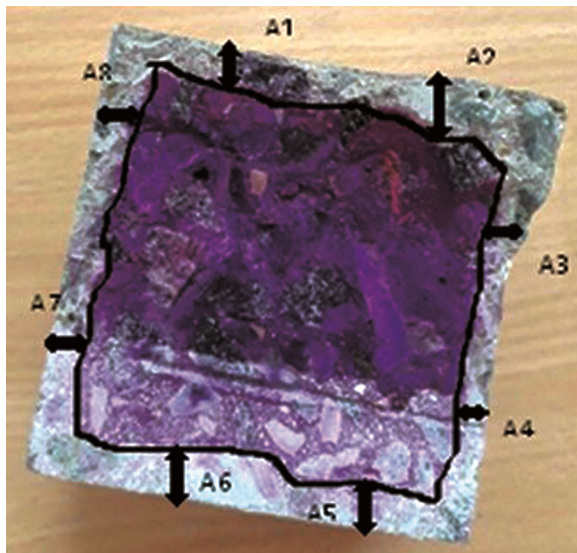


Fig. 2 — Carbonation depths analyzed from phenolphthalein indicator method.

carbonated. The existence of both peaks denotes that the specimen is partially carbonated.

Carbonation depth is obtained by analyzing and comparing the X-Ray diffraction pattern at different depths of concrete cubes. A significant reduction in the peak height of CaCO_3 can be observed with increasing the depth of the sample.

2.3.3 FTIR method

The Fourier Transform Infrared Spectroscopy (FTIR) method is a technique to determine the functional group present in a molecule. Every functional group vibrates in two directions, one along the bond (stretching frequency) while the other in the direction perpendicular to the bond (wagging frequency). Further, every atom possesses a unique stretching and wagging frequency. This helps to identify the specific atom/functional group present in a molecule. When the Infrared rays pass through the molecule, the functional group present in the molecule absorbs the IR rays of matching frequency. The FTIR spectrum as shown in Fig. 4 is graphically represented as percentage transmittance (%T) or percentage absorbance (%A) versus wavenumber. The type of bond/functional group is indicated by the wave number



Fig. 3 — XRD apparatus with model Mini Flex 600 (IIT- BHU Varanasi).



Fig. 4 — FTIR instrument (IIT- BHU Varanasi).

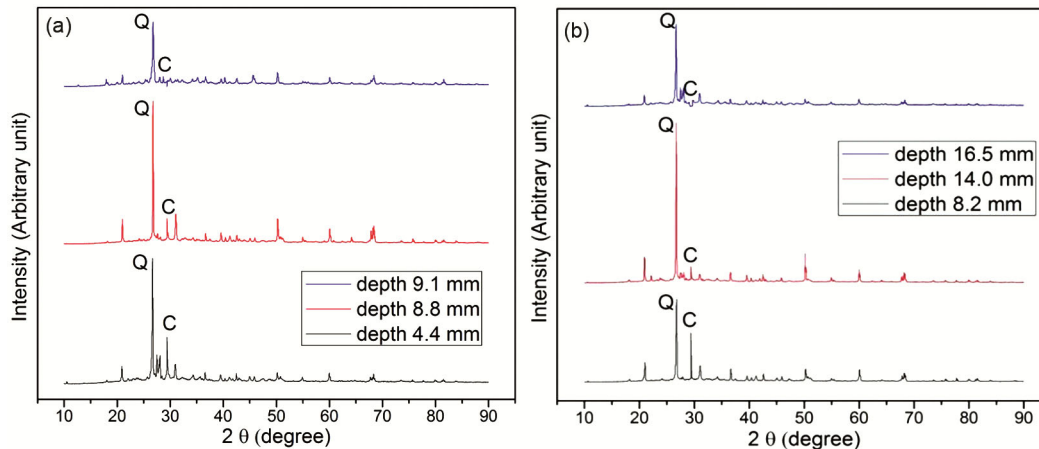


Fig. 5 — XRDA results for opc concrete at W/C 0.4 (a) OPC concrete and (b) PPC concrete.

of the peak. The graph falls in the transmittance curve while it peaks in the absorbance curve at a specific wave number or frequency which is indicative of carbonation depth. At the interface, the spectral changes associated with the presence of $\text{Ca}(\text{OH})_2$ have been identified by using infrared spectroscopy on concrete samples. The process of carbonation includes the conversion of $\text{C}=\text{O}$ bonds of CO_2 into $\text{C}-\text{O}$ bonds in the CaCO_3 having a range of wave numbers between $1410\text{--}1510\text{ cm}^{-1}$. Hence, the wave number of the $\text{C}-\text{O}$ bond indicates the carbonation in concrete.

3 Results and Discussion

3.1 Results of phenolphthalein indicator method

Results of carbonation depths of concrete samples are obtained at various sections and averaged to

conclude carbonation depths of concrete at variable W/C ratio. Carbonation depths of OPC concrete samples are found to be 4.4 mm, 7.5 mm, and 10.1 mm at W/C ratios of 0.4, 0.45, and 0.5 respectively. Carbonation depths of PPC concrete samples are found to be 8.2 mm, 14.1 mm, and 18.8 mm at W/C ratios of 0.4, 0.45, and 0.5 respectively.

3.2 Results of the XRDA method

Figures (5-7) show the XRDA Graph between intensity and diffraction angle (2θ) of different types of concrete at 3 days curing period, 4 % CO_2 concentration and 65 % relative humidity and retention period of 20 days in carbonation chamber for OPC concrete and PPC concrete at variable W/C ratio 0.4, 0.45 and 0.5. Q represents the peak value of intensity for quartz (SiO_2) and C represents the peak value of

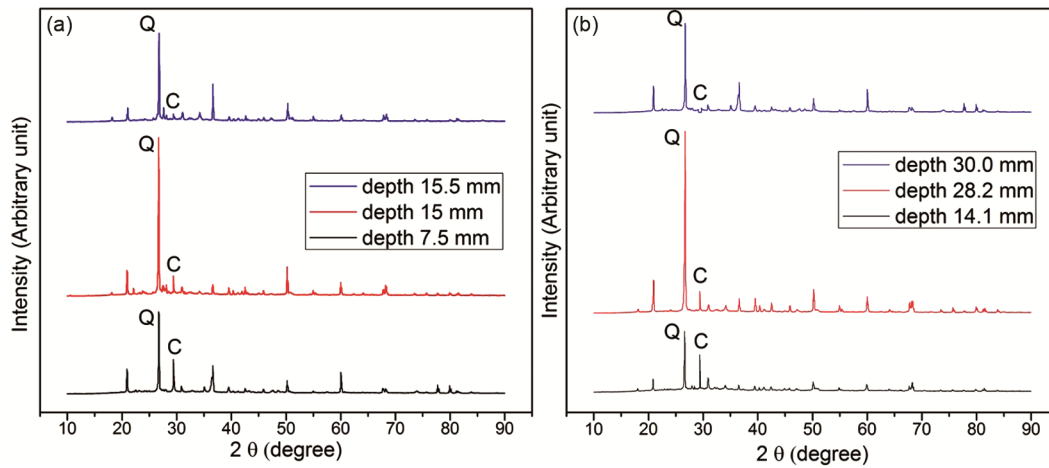


Fig. 6 — XRD results for opc concrete at W/C 0.45 (a) OPC concrete, and (b) PPC concrete.

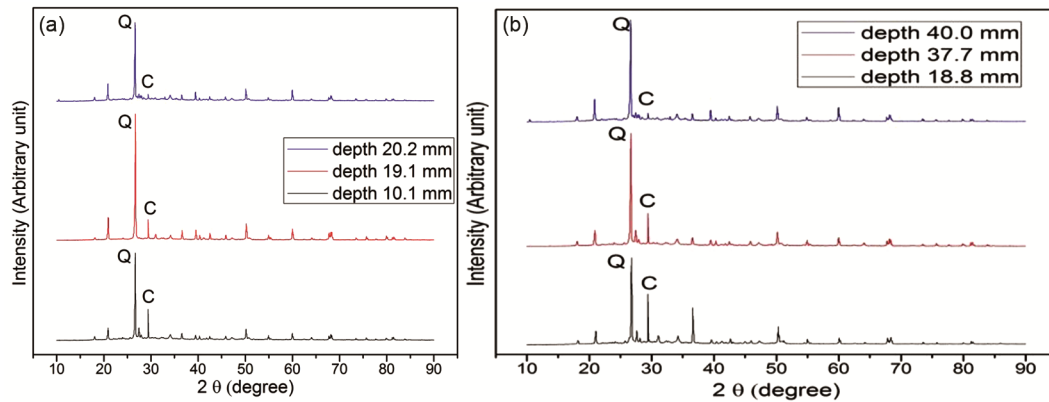


Fig. 7 — XRD results for opc concrete at W/C 0.5 (a) OPC concrete, and (b) PPC concrete.

intensity for CaCO_3 . The appearance of peak value for CaCO_3 in the graphs indicates carbonation depth.

From Fig. 5(a), it can be inferred that the depth of carbonation in the concrete sample of OPC cement with a 0.4 W/C ratio is 8.8 mm, since peak values of CaCO_3 are observed in graphs corresponding to 4.4 mm and 8.8 mm but the value is extremely small for graph corresponding 9.1 mm. For PPC concrete, XRD analysis of sliced concrete samples randomly at depths of 8.2 mm, 14.0 mm, and 16.5 mm for 2θ values corresponding to CaCO_3 (C) and SiO_2 (Q) represented in Fig. 5(b) reveals that the peak value of intensity for 2θ values corresponding to CaCO_3 is observed in graphs at 8.2 mm sample and 14.0 mm sample but not in 16.5 mm sample. Thus the depth of PPC concrete carbonation can be ascertained as 14.0 mm.

From Fig. 6(a), it can be inferred that the depth of carbonation in the concrete sample of OPC cement with a 0.45 W/C ratio is 15.2 mm, since peak values of CaCO_3 are observed in graphs

corresponding to 7.5 mm and 15.0 mm but the value is extremely small for graph corresponding to 15.5 mm. Hence, carbonation depth lies between 15.0 mm and 15.5 mm and is thus interpolated as 15.2 mm. For XRD analysis of sliced PPC concrete samples randomly at depths of 14.1 mm, 28.2 mm, and 30.0 mm for 2θ values corresponding to CaCO_3 (C) and SiO_2 (Q) represented in Fig. 6(b) reveals that the peak value of intensity for 2θ values corresponding to CaCO_3 is observed in graphs at 14.1 mm sample and 28.2 mm sample but not in 30.0 mm sample. Thus the depth of carbonation can be ascertained as 28.2 mm PPC concrete with 0.45 W/C.

From Fig. 7(a), it can be inferred that the depth of carbonation in the concrete sample of OPC cement with a 0.5 W/C ratio is 19.4 mm, since peak values of CaCO_3 are observed in graphs corresponding to 10.1 mm and 19.1 mm but the value is extremely small for graph corresponding to 20.2 mm. Hence, carbonation depth lies between 19.1 mm and 20.2 mm and is thus interpolated as 19.4 mm. XRD analysis of sliced PPC

concrete samples randomly at depths of 18.8 mm, 37.7 mm, and 40.0 mm for 2θ values corresponding to CaCO_3 (C) and SiO_2 (Q) represented in Fig. 7(b) reveals that the peak value of intensity for 2θ values corresponding to CaCO_3 is observed in graphs at 18.8 mm sample and 37.7 mm sample while the extremely small peak is observed at 40.0 mm. Thus the depth of carbonation lies between 37.7 mm and 40.0 mm. It can be inferred that the depth of carbonation is 38.8 mm.

3.3 Results of the FTIR method

In the FTIR method, Carbonation depths can be determined from a study of the specific peak of the C–O functional group in the wavenumber between $1410\text{--}1510\text{ cm}^{-1}$. The carbonation depth is found from the position of the C–O specific peaks relative to the baseline. Graphs between absorbance and wavenumber for a different type of concrete with different W/C ratio and the depth of carbonation is derived concerning a baseline at 37 % transmittance

as per Chang and Chen⁶. Peaks lying below the baseline indicate carbonation depth.

From Fig. 8(a), it can be inferred that the depth of carbonation in the concrete sample of OPC cement with a 0.4 W/C ratio is 9.4 mm, since the trough values lying in the range of Wave number $1410\text{--}1510\text{ cm}^{-1}$ falls below the baseline for each of the graphs about depths of 4.4 mm, 8.8 mm and 9.4 mm. Since 9.4 mm just lies at the baseline, hence the depth of carbonation is concluded at 9.4 mm. Fig. 8(b) shows that the carbonation depth in the concrete sample of PPC cement with 0.4 W/C is 16.3 mm since the trough values lying in the range of Wave number $1410\text{--}1510\text{ cm}^{-1}$ falls below the baseline for each of the graphs about depths of 8.25 mm and 14.0 mm and lies just above the baseline for the graph about 16.5 mm. Hence, it can be inferred that the depth of carbonation is 16.3 mm.

From Fig. 9 (a), it can be inferred that the depth of carbonation in the concrete sample of OPC cement with a 0.45 W/C ratio is 15.3 mm since the

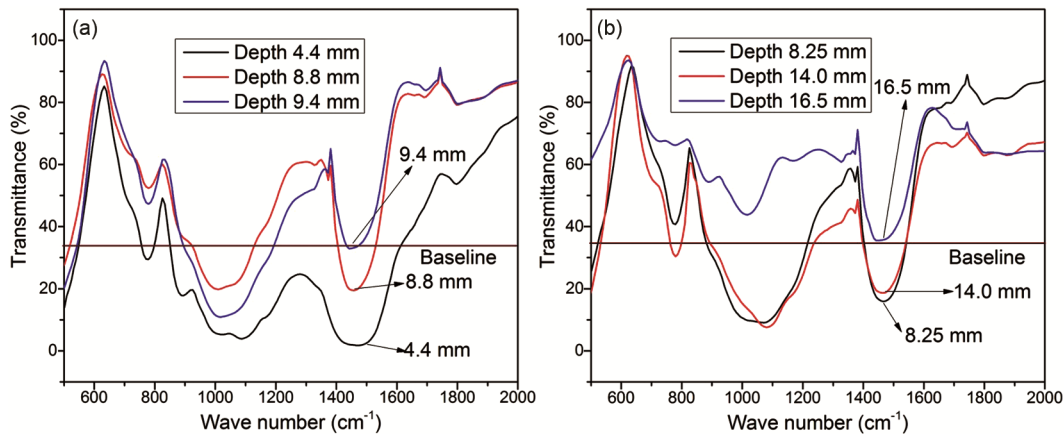


Fig. 8 — FTIR results for opc concrete at W/C 0.4 (a) OPC concrete, (b)PPC concrete.

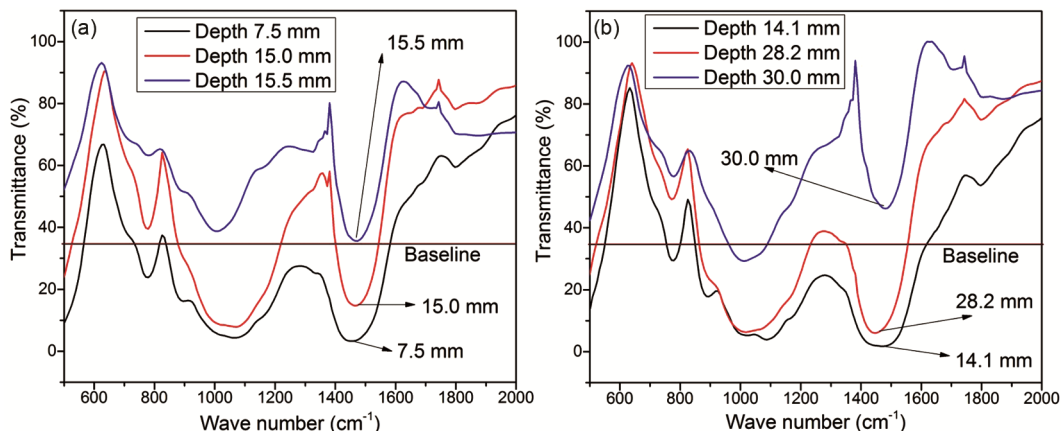


Fig. 9 — FTIR results for opc concrete at W/C 0.45 (a) OPC concrete, and (b)PPC concrete

trough values lying in the range of Wave number 1410–1510 cm^{-1} fall below the baseline for each of the graphs of depths of 7.5 mm and 15.0 mm and lies just above the baseline for the graph of 15.5 mm. Hence, the depth of carbonation is interpolated as 15.3 mm. Fig. 9(b) shows that the depth of carbonation in the concrete sample of PPC cement with a 0.45 W/C ratio is 29.4 mm since the trough values lying in the range of Wave number 1410–1510 cm^{-1} falls below the baseline for each of the graphs about depths of 14.1 mm and 28.2 mm and lies above the baseline for 30.0 mm. Since the baseline is in between the graphs for 28.2 mm and 30.0 mm, it can be inferred that the depth of carbonation is 29.4 mm.

From Fig. 10(a), it can be inferred that the depth of carbonation in the concrete sample of OPC cement with a 0.5 W/C ratio is 19.7 mm, since the trough values lying in the range of Wave number 1410–1510 cm^{-1} falls below the baseline for each of the graphs about depths of 10.1 mm and 19.1 mm and lies above the baseline for 20.2 mm. Since the

baseline is in between the graphs for 19.1 mm and 20.2 mm, the depth of carbonation is interpolated as 19.7 mm. Fig. 10(b) shows that the depth of carbonation in the concrete sample of PPC cement with 0.5 W/C is 34.0 mm since the trough values lying in the range of Wave number 1410–1510 cm^{-1} falls below the baseline for the graph of depths of 18.85 mm and lies above the baseline for each of the graphs of 36.0 mm and 37.75 mm. Since the graph corresponding to 36.0 mm depth is near the baseline, it can be inferred that the depth of carbonation is 34.0 mm.

3.4 Comparison of carbonation depth obtained from various methods

Comparison of carbonation depth of OPC and PPC Concrete obtained from Phenolphthalein, XRDA, and FTIR method are shown in Fig. 11. From Fig. 11, it can be inferred that the carbonation depth obtained from advanced methods XRDA and FTIR are much higher than those obtained from conventional

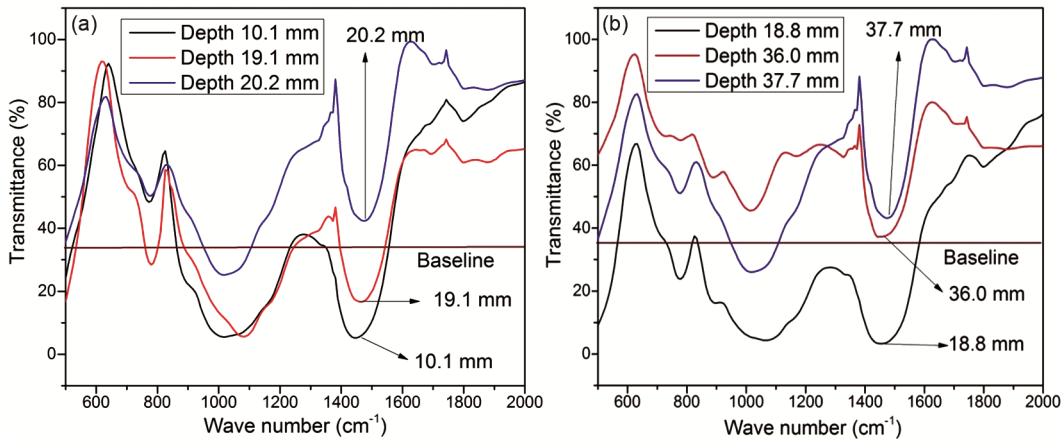


Fig. 10 — FTIR results for opc concrete at W/C 0.5 (a) OPC concrete and (b) PPC concrete.

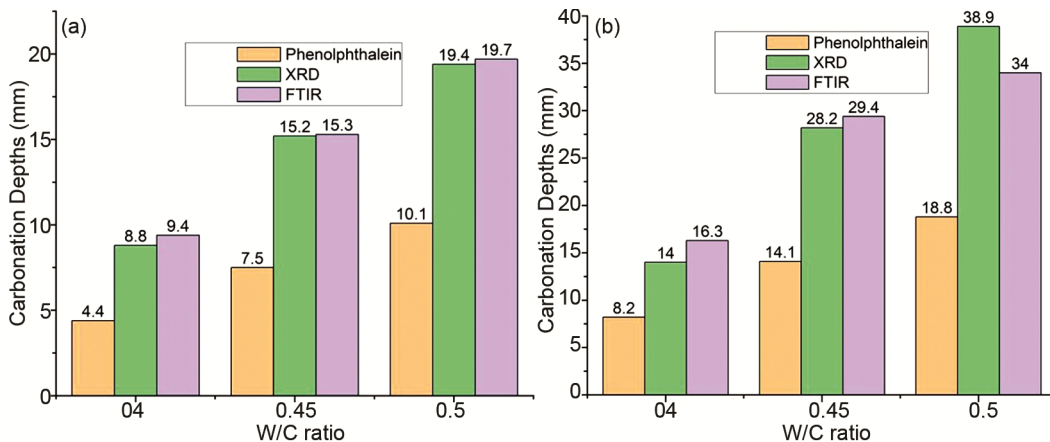


Fig. 11 — Carbonation depths of concrete obtained by various three methods (a) OPC concrete, and (b) PPC concrete.

Table 3 — Comparison of carbonation depths of OPC concrete by various methods

Mix	W/C	Carbonation depths (mm)			% Difference of carbonation depths by	
		Phenolphthalein	XRDA	FTIR	XRDA and Phenolphthalein	FTIR and Phenolphthalein
O ₁	0.4	4.4	8.8	9.4	100	113.6
O ₂	0.45	7.5	15.2	15.3	102.7	104
O ₃	0.5	10.1	19.4	19.7	92.1	95

Table 4 — Comparison of carbonation depths of PPC concrete by various methods

Mix	W/C	Carbonation depths (mm)			% Difference of carbonation depths by	
		Phenolphthalein	XRDA	FTIR	XRDA and Phenolphthalein	FTIR and Phenolphthalein
P ₁	0.4	8.2	14.0	16.3	70.7	98.8
P ₂	0.45	14.1	28.2	29.4	100.0	108.5
P ₃	0.5	18.8	38.9	34.0	106.9	80.9

phenolphthalein indicator method for OPC and PPC Concrete, while the carbonation depth obtained from advanced methods XRDA and FTIR are nearly similar results for OPC and PPC Concrete. It is also observed that the carbonation depth of PPC concrete was greater in comparison to OPC concrete which is obtained from the phenolphthalein indicator method, XRDA, and FTIR methods with variable W/C ratio. The results obtained by three methods indicate the rate of carbonation in OPC concrete is lower in comparison to PPC concrete. Hence OPC concrete is suggested against PPC to reduce the risk of carbonation in concrete.

Table 3 and Table 4 show a comparative study of carbonation depths obtained from three different methods for OPC and PPC concrete respectively with variable W/C ratios. It is observed that the carbonation depth of concrete samples increases with increasing W/C ratio. It is also observed that carbonation depth determined from XRDA and FTIR methods was found to be nearly twice in comparison to the phenolphthalein indicator method for OPC and PPC concrete.

4 Conclusion

The paper deals determination of carbonation depth of OPC and PPC concrete from the experimental study by using the phenolphthalein indicator method, XRDA, and FTIR methods. Also, experimental results obtained by both conventional and advanced methods have been analyzed to draw a comparison of the test methodologies. Given the experimental results, it may not be out of place to mention that, since in the natural environment the rate of carbonation is extremely slow, and it is difficult to measure the carbonation in an actual structure, with effective life being much longer than the scope of an experiment. Since the experiments are conducted in an accelerated chamber under controlled conditions, results are

analyzed to understand the gross nature of the impact of various factors and the trend of their influence approximately. On the basis of the experimental results, the following significant conclusions can be drawn from this study:

- XRDA and FTIR methodologies compute the existence of CaCO₃ at pH levels ranging from 8.3 to 11.5, whereas the Phenolphthalein indicator method detects the level of carbonation only up to a pH value of 9.5. Hence advanced methods are more suitable for detecting of carbonation depth of concrete in comparison to the phenolphthalein indicator method.
- Carbonation depth determined from XRDA and FTIR methods was found to be nearly twice the results obtained from the phenolphthalein indicator method for OPC and PPC concrete with variable W/C ratio.
- OPC concrete is recommended against PPC concrete to reduce the risk of carbonation in concrete due to the lower carbonation in OPC concrete in comparison to PPC concrete which is obtained by three different methods of phenolphthalein indicator method, XRDA, and FTIR.
- Depth of carbonation in concrete increases with the increase of the W/C ratio for both OPC and PPC Concrete.

LIST OF ABBREVIATIONS:

- XRDA = X-ray diffraction analysis
- FTIR = Fourier Transform Infra-Red
- OPC = Ordinary Portland Cement
- O₁, O₂, and O₃ = Mix the proportion of OPC concrete samples
- PPC = Portland Pozzolana Cement
- P₁, P₂, and P₃ = Mix the proportion of PPC concrete samples

- W/C = Water/Cement
- W = Water
- C = Cement
- FA = fine aggregate
- CA₁₀ = Course aggregate (10 mm)
- CA₂₀ = Course aggregate (20 mm)

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