

A Study for Carbonation Degree on Concrete using a Phenolphthalein Indicator and Fourier-Transform Infrared Spectroscopy

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Abstract—A concrete structure is designed and constructed for its purpose of use, and is expected to maintain its function for the target durable years from when it was planned. Nevertheless, as time elapses the structure gradually deteriorates and then eventually degrades to the point where the structure cannot exert the function for which it was planned. The performance of concrete that is able to maintain the level of the performance required over the designed period of use as it has less deterioration caused by the elapse of time under the designed condition is referred to as Durability. There are a number of causes of durability degradation, but especially chloride damage, carbonation, freeze-thaw, etc are the main causes. In this study, carbonation, one of the main causes of deterioration of the durability of a concrete structure, was investigated via a microstructure analysis technique. The method for the measurement of carbonation was studied using the existing indicator method, and the method of measuring the progress of carbonation in a quantitative manner was simultaneously studied using a FT-IR (Fourier-Transform Infrared) Spectrometer along with the microstructure analysis technique.

Keywords—Concrete, Carbonation, Microstructure, FT-IR

I. INTRODUCTION

CONCRETE structures structure undergoes a deterioration of properties and structural performances in the materials caused by cracking and deterioration/damage to the concrete owing to a variety of physical and environmental factors as time passes after the completion of construction. When such deterioration phenomenon is in progress and deepens, its safety, durability and functionality as the structure also deteriorates, which causes critical influences on the structural soundness. Concrete structure that is used by the public (bridge, harbor, etc) are found to have problems in part with regards to deterioration/damage, but there exists neither comprehensive data nor appropriate technologies concerning such a variety of deterioration factors and deterioration phenomena, and further the method for the evaluation of the structural soundness of a deteriorated structure is not yet fully established.

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Especially, concrete underground structure may face erosion through sulfuric acid, leaching, while the structure exposed to air is vulnerable to neutralization and chloride damage by incoming salt. In addition, the surface of the concrete wall body is vulnerable to damage by freeze-thaw owing to the repeated phenomenon of freeze thaw in winter as well as a relatively constant exposure to moisture[1].

Structures located on surface area face damages under fundamentally dry conditions, while deterioration is in progress under wet conditions if it exists under the surface. The deterioration acceleration test, as afore-mentioned, is essential for the evaluation of the actual degree of influence in regards to the concrete structure exposed to whole deterioration.

In this study, in order to establish the technology for evaluating the soundness of concrete structure in systematic and scientific manner and conduct active maintenance techniques, an experimental analysis was performed using concrete mix design and materials in such a manner through replacing fly ash for 20% of the carbonation caused by CO₂ in the air and other influential factors amongst deterioration damages. In regards to carbonation of the existing concrete structure, the boundary of color that changes when the indicator[2] is sprayed was considered as the depth of carbonation, which has been widely used. In this study, however, the existing method for measurement of the depth of carbonation using phenolphthalein and simultaneously the FT-IR (Fourier-Transform Infrared Spectrometer) was used to measure the depth of carbonation depending on the infiltration of the deterioration factor of carbonation from which, a certain difference was identified.

II. EXPERIMENTS

A. Procedure

Concrete bears a strong alkali at the time when it is manufactured (pH=12~13), which is due to the creation of large amounts of Ca(OH)₂ and such remains in concrete when the cement mineral in concrete reacts with water resulting in an aqueous solution. Such Ca(OH)₂ does not contribute to strength development, but forms passive protective oxide film, which plays an important role for corrosion prevention of the steel rebar inside the concrete[3]. The process that such concrete is neutralized is conducted in such a manner that light acid carbon gas in the air (approx. 0.035%) infiltrates through capillary pores on concrete surface and contacts the pore solution in the concrete where Ca(OH)₂ is dissolved and in turn changes into stable calcium carbonate and water. In this process, the reaction product as shown in Table 1 is created but the calcium carbonate is deposited on the pore wall inside concrete causing the

concrete tissue to increase in density, while water evaporate or is used in the chemical reaction continuously. Especially, when concrete is sufficiently wet and the water existing in the concrete pores flow out to the surface from the inside, calcium carbonate or alkali carbonates, that is, NaCO_3 , KCO_3 , etc. emerge outwardly together, which are then deposited little by little continuously on concrete surface resulting in efflorescence in the shape of white water flow. Calcium carbonate deposited on the pore wall inside concrete bears neutrality, where, depending on the degree where the Ca(OH)_2 is changed into calcium carbonate, pH is changed from a strong alkali to neutral gradually from the surface where carbon gas and water infiltrates easily. Through such process of neutralization, concrete neutralization accelerates in concrete with a high water-cement ratio creating large pores and low waterproofing properties from the aspect of concrete quality, and also in the place of high-concentration carbon gas and proper humidity (50~75%) from the environmental aspect. When the humidity is overly high, such blocks the internal pores of concrete that is the path for carbon gas to flow in rather affecting the neutralization. That is, neutralization occurs at a rapid speed in the environment where dry and wet conditions repeatedly occur.

Concrete carbonation phenomenon creates an environment that corrodes the steel rebar in the concrete. In such environment, corrosion may occur through the onslaught of chloride ions although a passive protective oxide film is formed[4]. As explained above, steel rebar can corrode through a decrease of the pH of the concrete close to the steel rebar. Pourbaix[5] reported that passive protective oxide film on the surface of steel rebar in concrete begins to corrode at the degree of pH at 10.4 or less. A pH level at 10.4 or less of the concrete that has strong alkali content means that water and oxygen has already sufficiently flowed owing to carbonation[6]. In general, as shown in Table 1, calcium carbonate and water are known to be created through carbonation. When carbonation is in progress up to the depth of the steel rebar, the steel rebar can be anticipated to have been exposed to a corrosive environment. Parrott reports that the supply of moisture and oxygen is an important element to corrosion by neutralization, and therefore neutralization occurs however no corrosion does at a relative humidity of less than 60%. Steel rust normally appears in such a manner that the original volume expands to 2.5 to 7 times resulting in cracking inside concrete, which causes severe durability degradation of a structure such as bond strength degradation, destruction of concrete, reduction in the cross sections of the steel rebar, etc. For this reason, it is important to determine the carbonation from the aspect of durability.

TABLE I
CONCRETE CARBONATION PRODUCTS

Portland cement hydration product	Carbonation products
Calcium hydroxide	Calcite and water
Calcium silicate hydrate	Calcite, silica gel, and water

Calcium aluminate hydrate	Calcite, alumina gel, and water
Hydrated ferrite phases	Calcite, ferric oxide, alumina gel, and water
Etringite and calcium monosulfoaluminate	Gypsum, alumina gel, and water

B. Production of specimen

The results of having analyzed the components of the binder used is as shown in Table 2, which shows no great difference as compared with those of Portland class I, and even compared with the result of Bogue's Equation. For the paste specimen of carbonation purpose, the researcher produced a cubic-shape specimen of 50×50×50mm under ASTM C 109. For all specimens used in this study, the researcher produced such with fly ash at a replacement ratio of 20% of OPC (Ordinary Portland Cement) in mass. In order to determine the change in the depth of carbonation depending on the W/B ratio, W/B was altered to 0.40(Mix 1), 0.45(Mix 2), and 0.50(Mix 3), and then Paste, Mortar, and Concrete specimens were produced in 3 pieces in each test for a total of 155, with which, the change in depth was measured. In the case of FT-IR analysis, the analysis was conducted using a paste specimen in order to limit the change in the result value owing to aggregate. All specimens were cured in water for 28 days at a temperature of $20 \pm 2.5^\circ\text{C}$ prior to the exposure of CO_2 .

TABLE II
RESULT OF ANALYSIS FOR THE COMPONENTS OF BINDERS USED

Components	OPC	FA
SiO_2	20.10	54.70
Al_2O_3	5.22	24.80
Fe_2O_3	2.76	8.76
CaO	55.90	2.99
MgO	2.97	1.15
SO_3	1.87	0.32
LOI	1.56	3.56

C. Accelerated carbonation test (Condition & method)

The carbonation test was performed in such a manner for the measurement of the depth of carbonation for each specimen using the acceleration tester preset to the temperature of 20°C , humidity of 60%, and CO_2 concentration of 5% with the ages of 28, 56, 91, 180, and 365 days respectively. All changes in the depth of carbonation were measured depending on the types of the specimens, such as paste, mortar, and concrete. Especially, in order to induce smooth carbonation of all specimens at the time of carbonation acceleration, a sufficient distance between each specimen was secured to ensure that the contact of the specimen to the air could be smoothly maintained.

D. Measurement of depth of carbonation

The existing method for the measurement of the depth of carbonation of concrete depends on colorless phenolphthalein indicator, which is sprayed onto concrete surface showing a change in color depending on the pH factor. Such indicator method depends on pH change only and therefore the influence may not be clearly identified if carbonation only partially occurs, or if carbonation occurs but with pH beyond the scope changeable by indicator. Especially, in the case of recent non-cement type materials with the pH of the material itself less than 9, there exists a limitation for the measurement of the depth of carbonation using such phenolphthalein indicator. Nevertheless, such simplest and easiest methods in a visual aspect for testing carbonation is quite widely used at construction sites, and its use may not be restricted in reality. This study focused on the identification of the differences between the two methods, that is, the method for the measurement of the carbonated depth through the production of a concrete specimen and then using the existing indicator through the accelerated carbonation test and the method the measurement of carbonation through microstructure analysis.

In measuring the carbonated depth using the indicator method in this study, the specimen in the carbonation chamber was removed at each test date and split with a universal testing machine. Then phenolphthalein solution was sprayed onto the surface area of the split specimen. The used indicator in this case was made in such a manner of having dissolved a 1% phenolphthalein solution in 99% ethanol and added distilled water thereto. The indicating range of phenolphthalein existed within the range of pH 8.3~9.5, and the pH range varied depending on the pH indicator as shown in Table 3.

In the case of concrete core extracted from the structure of low quality concrete and excessively elapsed duration, such tends to be difficult to judge the depth of neutralization from the cut area as time elapses from the date when the core was extracted. That is, it is difficult to observe the change in color as the micro-granule powder occurring during the extraction of the core remains on core surface or in the pores. Therefore, it is desirable to spray phenolphthalein solution to the cut area on the site immediately after exposure when the specimen is exposed to air to measure the carbonation depth at the time of extraction or testing the core so as to measure the depth of neutralization. If it is not possible to measure such on the site unavoidably, it is necessary to cut the center of specimen and then measure the area after spraying phenolphthalein solution onto the cut area of the concrete.

In this study, neutralization was measured at the cut area in the center of the specimen. In result, the area where neutralization was implemented did not show any change in the color of concrete while the alkali area had changed into red. The depth of the neutralized area was measured with the use of vernier calipers for max. depth, min. depth, and average depth. The average value was calculated based on three specimens as per the period exposed to carbonation was measured,

TABLE III
PH INDICATION VALUE OF SEVERAL INDICATORS

Indicator	pH range
Tropaeolin O	pH 11.1-12.7
Alizarin yellow R	pH 10.2-12.2
Thymolphthalein	pH 9.3-10.5
Phenolphthalein	pH 8.3-9.5

E. Results of test

The results of the measurement on the change in depth of carbonation using the indicator method are as stated in Table 4. After the specimen was produced, such were cured for 28 days underwater, and exposed to Accelerated Concrete Carbonation Tester, and then carbonation was measured for five stages, such as the ages of 28, 56, 91, 180, and 365 days. The test was performed in such a manner of splitting three specimens by each age and spraying phenolphthalein solution onto the center, and measuring the distance from the surface to the color-changed area using vernier calipers. The average value was calculated based on three each specimens by ages and types. The following Fig. 1 - 3 show the depth of carbonation of Paste, Mortar, and Concrete by Mix depending on the type of specimen. Fig. 4 shows a photograph of the process indicating the change in the depth of carbonation of Mix 1 using the indicator method. According to the analysis of the results in Fig. 1 - 3, as W/B increases, the depth of carbonation tends to increase, which is possibly due to the environment where carbon ion could deeply infiltrate the specimen as the number of capillary pores increased caused by the increase of the W/B ratio. The width of change in W/B was changed equally, but there were rare changes of Mix 2 and Mix 3 in the case of paste and mortar. That is, the existence of critical W/B for the control of the speed of carbonation could be anticipated, but it could not yet be verified. In the case of concrete, only Mix 3 showed an increasing ratio of carbonated depth in a large width. The relation between the inflow/outflow of carbon ion through the surface of coarse aggregate and W/B may be part of the anticipation, but might not be mentioned further as it could not be verified. The part that can be mentioned with confidence in this test result is that the depth of carbonation increases as the W/B increases. This test result will be used as basic data for the test results of carbonated depth using the existing indicator method, and then a comparison will be made to the test result for the carbonation application analysis technique later.

TABLE IV
CARBONATION DEPTH BY DURATION

Mix Type	depth(mm)	28	56	91	180	365
		days	days	days	days	days
Mix 1 (W/B 0.40)	Paste	0	0	8.50	14.50	16.00
	Mortar	3.92	7.02	8.47	8.60	9.63
	Concrete	8.52	10.63	14.88	17.64	18.30

Mix 2 (W/B 0.45)	Paste	0	3.65	10.52	21.09	25.00
	Mortar	4.88	11.94	16.61	20.75	25.00
	Concrete	7.00	12.99	15.00	19.99	23.20
Mix 3 (W/B 0.50)	Paste	0	7.40	10.83	16.62	25.00
	Mortar	6.83	11.94	16.61	20.75	25.00
	Concrete	12.33	21.52	29.56	36.40	41.10

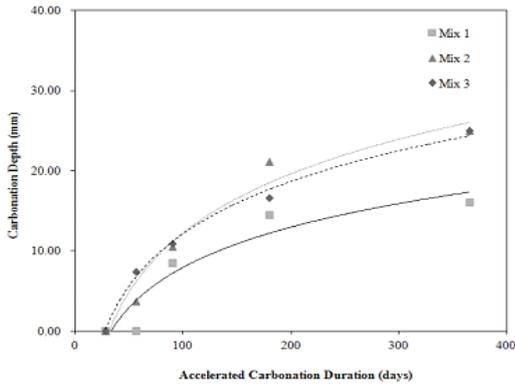


Fig. 1 Paste

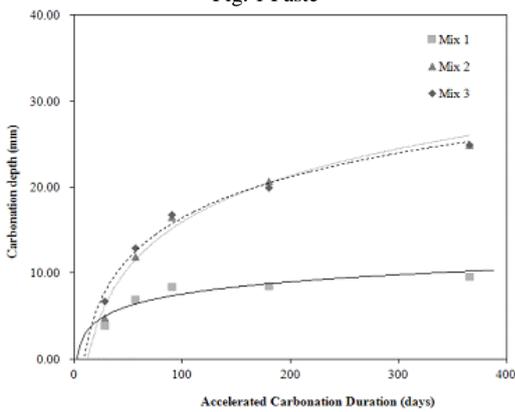


Fig. 2 Mortar

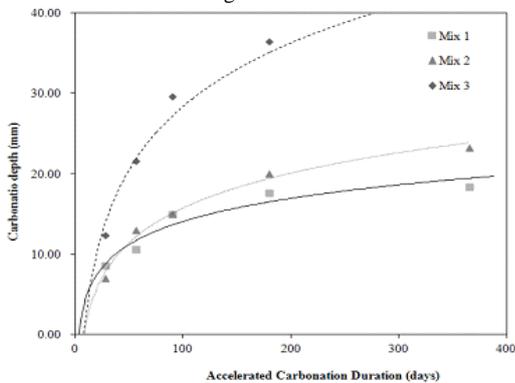


Fig. 3 Concrete

III. RESULT OF ANALYSIS

A. Procedure

This analysis was made for the comparison between the results of testing carbonated depth using the existing indicator method and the results of testing the progress of carbonation using the FT-IR analysis machine.

Fig. 5 shows the results of the FT-IR calibration curve according to the content ratio of calcium carbonate and Ca(OH)_2 as the back data for seeking the conversion of Ca(OH)_2 for quantitative and qualitative analysis using FT-IR. In general, the range of Concrete Band is as stated in Table 5. Fig. 5 shows the FT-IR peak based on the calcium carbonate- Ca(OH)_2 ratio. Carbonate bands peak was measured at $930 - 730\text{cm}^{-1}$, $1833 - 1782\text{cm}^{-1}$ and $2646 - 2423\text{cm}^{-1}$ where more intensity was displayed as the contents of calcium carbonate were increased. The Ca(OH)_2 spectrum showed a strong O-H peak. As the content of calcium carbonate increases, O-H peak weakens and then altogether disappears in 100% calcium carbonate. Therefore the fraction of calcium carbonate through the integration of a certain peak ($879 - 711\text{cm}^{-1}$) using the FT-IR enabled a quantitative analysis of calcium carbonate in the mix of calcium carbonate and Ca(OH)_2 [7].

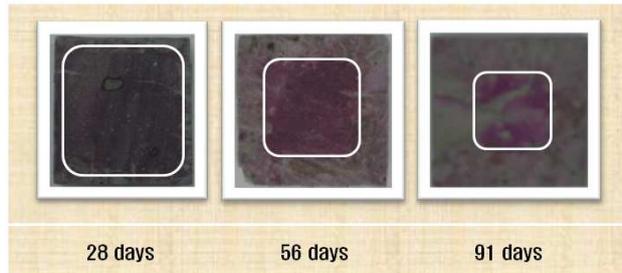


Fig. 4 Change in carbonation using phenolphthalein solution

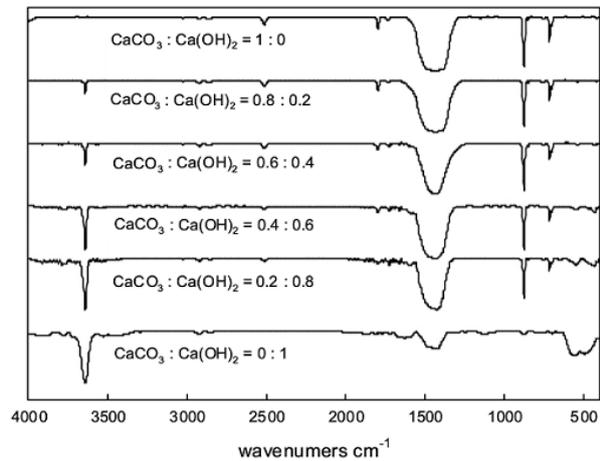


Fig. 5 FT-IR peak of percentage $\text{CaCO}_3/\text{Ca(OH)}_2$ mixture[7]

TABLE V
COMPONENTS ACCORDING TO THE RANGE OF FT-IR BANDS

Bands Identification		Bands Identification	
cm	group	cm	group
3640	OH	3696	OH
3402	S-O	3620	CH ₂
1622	S-O	3380-3450	OH
1426	CO ₃ ²⁻	2940-2864	CH ₂ ,CH ₃
1136	S-O	1740	C-O
920	Si-O	1442	CH ₂ ,CH ₃ , CO ₃ ²⁻
880	Si-O;Al-O;CO ₃	1378	C-CH ₃
746	Al-O	1244	C-O
712	CO ₃ ²⁻	1102	C-O, OH
660	S-O	1022	C-O,CH ₃
558	Si-O	944	C-C in ester group
522	Si-O;Al-O	882	CO ₃ ²⁻
448	Si-O;Al-O	748	CH ₂ ,CO ₃ ²⁻
		630	OCO
		606	C-O;ester group
		540	C-O

B. Analysis condition

For the carbonation analysis by depth, the paste powder was extracted at 5mm intervals from the specimen surface. After mixing the paste powder in 20mg and KBr in 200mg, such were formed into pellets using a high-pressure injection molding machine. Bruker's Bruker IFS-66/S was used as the FT-IR testing equipment where the analysis condition was a scan frequency of 32 times/sec, resolution of 4cm⁻¹, range of measurement of 400 - 4000cm⁻¹

C. Analysis result

Fig. 6 shows the result of FT-IR analysis measured at the specimen surface for 28 and 365 days of Mix 1. As shown in Table 5, the range of band by components can be divided into the following; calcium hydroxide OH group bands in 3642cm⁻¹, C-S-H bonding in 3440 - 3446cm⁻¹, sulfate phases band in 1116 - 1118cm⁻¹, anhydrous calcium silicates bands in 920, 526 - 536, and 458 - 464cm⁻¹ while carbonate phases in 1424 - 1436, 874 - 880, and 704 - 712cm⁻¹ in part. Especially for the analysis result of exposure to carbonation for 28 days and 365 days, a remarkable phenomenon was the extensive decrease of 3640 - 3696cm⁻¹ in the range of band related with Portlandite and the significant increase of 1426 - 1740 and 1022 - 1244cm⁻¹ in the range of band related with Calcite.

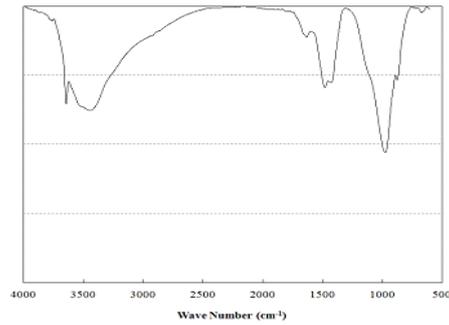


Fig. 6 (a) FT-IR Analysis Result for Mix 1 28 days

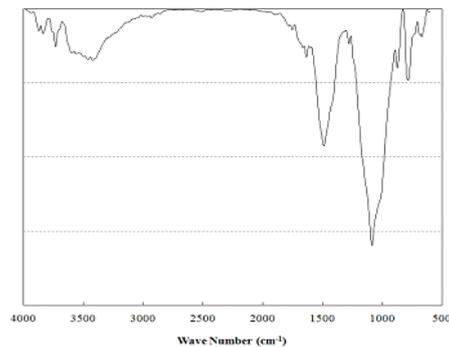


Fig. 6 (b) FT-IR Analysis Result for Mix 1 365 days

The analysis results in this study seems similar to the results of FT-IR based on the ratio of Portlandite and Calcite in Fig. 5 as aforementioned. Therefore, reflecting the expression of the Calcite-Portlandite ratio in Fig. 5, the influence of carbonation was expressed with Calcite/Portlandite with the assumption that the deteriorated material owing to the carbonation of Portlandite changes into Calcite. Studies on the quantification of deteriorated materials using FT-IR are widely conducted now, and a variety of methods are currently under development. However, for the nature of FT-IR analysis, for reasons of reduction in data recurrence following the stability of a light source, reduction in the efficiency of a beam splitter owing to humidity, the quantification of analysis result is difficult in reality as compared with the analysis equipment using the mass such as TG-DTA[8]. Therefore this study adopted the method of relatively evaluating the volume of Calcite and Portlandite that exists in the same specimen regardless of the time and environment of analysis in order to supplement such problems and showed the degree of carbonation using the method of calculating the ratio of intensity in the band range of Calcite and Portlandite depending on carbonation. That is, the result of the FT-IR analysis for a depth of 5mm from the surface is shown in Table 6. As stated in Table 6, as carbonation is in progress, it shows a quite meaningful value in the Calcite/Portlandite intensity ratio.

TABLE VI
CALCITE/PORTLANDITE USING INTENSITY OF FT-IR

Calcite/Portlandite	28 days	56 days	91 days	180 days	365 days
Mix 1	0.570	0.602	0.632	0.646	0.660
Mix 2	0.576	0.603	0.631	0.632	0.681
Mix 3	0.565	0.617	0.652	0.652	0.688

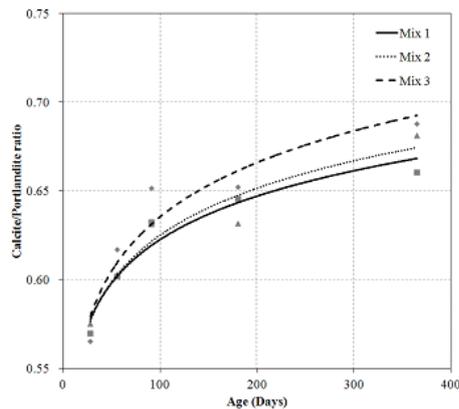


Fig. 7 Calcite/Portlandite using the intensity of FT-IR

Fig. 7 shows the result value of Table 6 in the graph expressing tendency, which indicates a similarity to the graph on the increasing depth of carbonation using the indicator method of Fig. 1 - 3. Nevertheless, the graph using the indicator method in Fig. 1 - 3 tends to increase in the depth of carbonation depending on the elapsed time, but the graph of Fig. 7 shows an increase of 5mm in carbonation from the surface, and therefore the meaning can be said to differ from each other. Nevertheless, if the features of Calcite/Portlandite owing to time lapse can be re-analyzed based on the relation of pH change, amongst the depths of carbonation of Mix 3 of paste specimen cured for 56 days under the indicator method, only the one that exceeded 5mm of analyzed depth was 7.40mm. The method of determining the carbonation using FT-IR by the afore-mentioned result could be an absurd prediction due to the adoption of insufficient data, but it is very encouraging from the aspect figures that the figure using the precise analysis method rather than a simple depth measurement method can be suggested using Calcite/Portlandite.

IV. CONCLUSION

This study aimed at the method of measurement about the progress of carbonation using the indicator method that is currently used and FT-IR that is one of a microstructure analysis technique performed simultaneously in order to evaluate the progress of carbonation of the concrete structure caused by carbonation amongst the deterioration phenomena of concrete structures. The specimens in the forms of paste, mortar, and

concrete were all produced by us, and each specimen was used in such a manner that fly ash 20% was replaced based on OPC in view that it is a widely used mixing ratio as the use of fly ash is on the increase. In addition, in order to identify the change in the degree of carbonation based on W/B, the test was implemented with W/B divided into three stages of 0.40, 0.45, and 0.50. As a result, as W/B increased, the depth of carbonation appeared remarkably. In the cases of Paste and Mortar, there was no significant difference in the speed of increase in the depth of carbonation of Mix 2 and Mix 3, while only Mix 1 showed a relatively high level of resistance against carbonation. On the other hand, in the case of the concrete specimens, the speed of carbonation of Mix 3 increased, which represented the effect of increase in the speed of carbonation caused by coarse aggregate[9].

As a result of analyzing FT-IR implemented together with the indicator method, it was found that the intensity of Portlandite decreased owing to carbonation while the intensity of Calcite increased. Based on the results of existing studies and this study, it was possible to suggest a method for predicting the progress of carbonation using the ratio of Calcite/Portlandite. Nevertheless, the results of the testing and analysis in this study were insufficient, and therefore require further supplementary testing, and the method of analyzing and predicting the progress of carbonation through the application of the latest analysis techniques together with the other micro-structure analysis techniques concurrently.

ACKNOWLEDGEMENT

This work was supported by the Energy Efficiency and Resources of the Korea Institute of Energy Technology Evaluation and Planning (KETEP) grant funded by the Korea government Ministry of Knowledge Economy (No. 2011T100200161)

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