Pergamon

Cement and Concrete Research, Vol. 28, No. 10, pp. 1379–1391, 1998

0008-8846/98 $19.00 + .00

PII S0008-8846(98)00076-3

# CHARACTERIZATION OF PORTLAND CEMENT CONCRETE USING ELECTROMAGNETIC WAVES OVER THE MICROWAVE FREQUENCIES

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(Received July 11, 1997; in final form May 11, 1998)

## ABSTRACT

A study was conducted to evaluate the effect of the basic properties of Portland cement concrete (PCC) and chloride presence (in PCC) on its dielectric properties over the microwave frequency range of 0.1 to 1 GHz. PCC specimens were prepared at three w/c ratios, 0.35, 0.45, and 0.55, using three aggregate types, limestone, quartzite, and active-silica, and two types of cement, type I and high alkali. Other PCC mixes were prepared and contaminated with two methods, addition of sodium chloride (NaCl) to the mixing water and immersion of hardened PCC specimens (prepared at a 0.45 w/c ratio) in 6% (by wt) NaCl solution after 28 days of curing. The dielectric properties were evaluated using a coaxial transmission line designed to allow measurements over the frequency range of 0.1 to 1 GHz. The dielectric properties of PCC specimens were evaluated over curing time. Powdered PCC samples were obtained from chloride contaminated specimens and chloride contents were determined using Virginia Tech Procedure. The results showed significant effects of curing time on the dielectric properties of PCC specimens and insignificant effects of w/c ratio, air entrainment, and cement type. The dielectric properties showed a significant difference between PCC specimens with different aggregate types. The chloride presence (in PCC) did not influence significantly the PCC dielectric properties. *© 1998 Elsevier Science Ltd*

## Introduction

Portland cement concrete (PCC) is a widely used construction material because of its abundance, ease of preparing and molding, and the low price of its constituents (aggregate and Portland cement). Properly designed, consolidated, and cured PCC with adequate cover to steel reinforcement will normally give satisfactory performance in service. However, lack of quality control in the field sometimes yields PCC with high porosity and low strength. This

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allows water loaded with chlorides to penetrate its internal structure, resulting in chemical activities that contribute to weakening and deterioration of the structure. The deterioration caused by reinforcing steel corrosion leads to cracking, then spalling of PCC. This requires reactive maintenance and repair if not detected at an early stage. Previous experience (1,2) showed that repair and maintenance procedures were not only expensive, but also ineffective because of a lack of knowledge of the deterioration-causing factors. Knowledge of the nature of the causative factor helps identify the most appropriate repair techniques and results in ending the problem and saving millions of dollars.

Different nondestructive techniques have recently been developed or utilized for the purpose of determining the cause of different deterioration in PCC. These include ultrasonic and infrared waves, three electrode linear polarization (3LP), and impact echo (3,4). However, limited success has been reported using these techniques because many of them detect deterioration at late stages rather than determine the causative factor. Recently, the utilization of electromagnetic (EM) waves in studying the physical and chemical changes in hydrating cement and detecting deterioration in pavements and bridges has been introduced.

Electromagnetic waves at 1 KHz were used to study the cement paste setting stages through the evaluation of the change in the electrical properties response (5). On the other hand, electrical conductivity and resistivity were used to study the cement paste at ages range from the mixing time to 24 h (6–10). The mechanical properties of a mature cement paste was studied, using both the dielectric properties and conductivity of the cement matrix constituents to estimate the volume of each constituent (11). Wittmann and Schlude (12) investigated the effect of humidity and temperature change on the complex permittivity of cement paste over frequencies range from 8.5 to 12.3 GHz. The study showed that the evaporable water change is very decisive in altering the complex permittivity value, where chemically bound water change does not affect the overall value.

Ground penetration radar (GPR) was used in the field to inspect concrete structures. It was used to detect and estimate voids underneath jointed concrete pavements, and to detect chloride content and/or deterioration in bridge decks (13–15). However, the success achieved in this regard was limited due to the following factors: first, the GPR systems used did not provide high penetration and resolution, due to some limitations such as multiple reflections and lack of a clear wave signal because of the heterogeneous dissipating nature of PCC; second, there was a lack of understanding of the physical and chemical properties of PCC on its dielectric properties; third, there was also a lack of understanding of the effect of chloride presence on PCC dielectric properties. The latter two factors have influenced the interpretations of GPR signals acquired after testing deteriorated and chloride contaminated PCC structures. This is because the processing of the signal depended on estimating the dielectric properties values without taking into consideration the effect of PCC ingredients, its moisture content, or chloride presence (in PCC).

As can be seen, there is a need to establish an understanding of the effect on PCC dielectric properties of changes in its basic properties and exposure to chloride over the microwave frequencies. This would allow better interpretation of GPR measurements. In addition, it would help assigning (if any) the most appropriate microwave frequencies appropriate for characterization of PCC basic properties, and/or detecting chloride contamination through evaluating its dielectric properties. Therefore, an experimental program to investigate the effect of PCC basic properties and chloride presence on its dielectric properties was conducted over the microwave frequency range of 100 MHz to 1 GHz, using a coaxial transmission line (CTL). This setup allows ranges of frequency that can’t be achieved using other simpler EM setups. The CTL is to be used in laboratory to measure, precisely, the dielectric properties of PCC which would be used later to interpret GPR measurements: this setup is not by any means a substitute of the GPR.

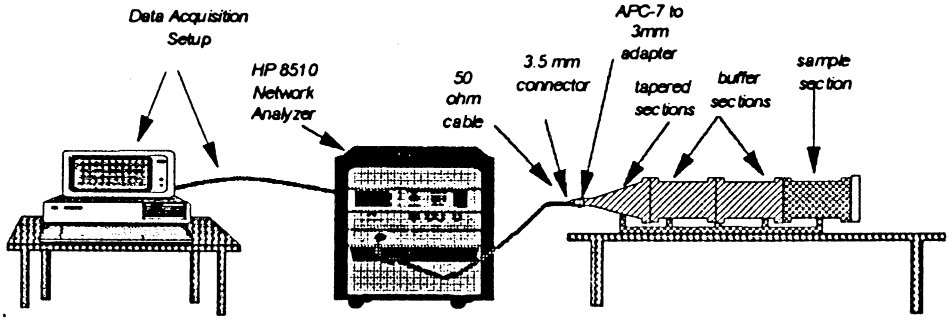
To study the effect of PCC's basic properties on its dielectric properties, PCC mixes were prepared at three w/c ratios, 0.35, 0.45, and 0.55, using three types of aggregate, limestone, quartzite, and active silica (AS) with and without air entrainment. Two types of cement were used, Type I and high alkali Portland cement. The dielectric properties were evaluated over curing time, from 1 to 28 days. The effect of chloride presence on the dielectric properties of PCC specimens was studied, using PCC mixes prepared at a 0.45 w/c ratio with quartzite and limestone aggregate without air entrainment. Two methods of chloride contamination were evaluated, partial immersion in 6% (by wt.) NaCl solution and addition of NaCl to the mixing water. The dielectric properties were evaluated over time.

## Complex Dielectric Properties (Constant)

The exposure of a dielectric material (not a perfect conductor) to an electromagnetic (EM) field results in changing the arrangements of its microscopic electric dipoles composed of positive and negative charges whose centers do not quite coincide. These are not free charges, and they cannot contribute to the conduction process. Rather, they are bound in place by atomic and molecular forces and can only shift position slightly in response to external fields. Free charges, on the other hand, are the ones that determine conductivity. Upon the exposure to EM, a shift in the relative positions of the internal bound positive and negative charges against normal molecular and atomic forces results in a storage of an electric energy in what is known as polarization. The latter is expressed by the complex dielectric properties (or constant) of the material. While the real dielectric constant reflects the amount of polarization of the material, the imaginary part reflects the losses caused by conductivity (controlled by free charges) and the relaxation of the water dipole. This is why a perfect dry dielectric (with no free charges) would have an imaginary part of zero. The possible polarization forms in PCC, at the radio wave frequencies, are caused by a combination of ionic, electronic, dipolar, and heterogeneous polarization. At the microwave frequencies, however, the contribution of dipolar polarization is much less, and heterogeneous polarization is absent. Yet, losses due to water relaxation (time until the water dipole fully orients) is increased, while conductivity losses are dramatically reduced. Therefore, changes in concrete chemistry (change in the composition of cement and/or aggregate), water content and state, and/or ions' content (in pore water) would result in changing its complex dielectric properties.

## Coaxial Transmission Line Measurements

A CTL was designed to characterize PCC's dielectric properties over a frequency range of 100 MHz to 1 GHz (Fig. 1). PCC specimens, 15 cm in diameter and 15 cm long with a centered 2.5-cm-diameter brass rod, were used to measure the PCC's complex dielectric properties. Measurements were performed in the time domain by employing time reflectometry or transmission (TDR or TDT) principles according to the theory presented in Al-Qadi



a. General Setup

b. Details of the CTL Components;
(a) Buffer Sections and (b) Tapered Section

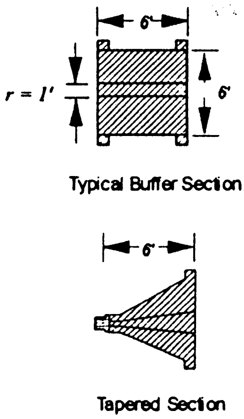


FIG. 1.
Setup of the coaxial transmission line.

et al. (16). The obtained dielectric properties showed a slight increase at the frequency of about 500 MHz that may be attributed to the step geometry imperfections. A corrective de-embedding process was performed to account for the systematic error related to the CTL design, therefore rectifying the approximate time domain measurement (17). Accuracy measurement was verified using a cylindrical Teflon specimen (with known dielectric constant). The repeated measurements showed that the CTL was able to measure the dielectric constant with an accuracy of 1–7.5% in the tange of 100 to 900 MHz. Due to the inconsistency of measured dielectric constant at the upper and lower frequency ranges, due to limitation of the CTL setup, the dielectric properties were evaluated on the frequency range of 261 to 750 MHz.

## Experimental Program

### Materials

In studying the effect of basic properties and chloride presence (in PCC) on PCC dielectric properties, three types of aggregate, limestone, quartzite, and AS, with Type I and high alkali

TABLE 1
Chemical composition of PCC components.

| Material | Compound weight (%) | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | $SiO_2$ | $Al_2O_3$ | $Fe_2O_3$ | CaO | MgO | $SO_3$ | $Na_2O$ | $K_2O$ | $TiO_2$ | O.O. | L† |
| HA* | 20.44 | 3.72 | 2.76 | 62.82 | 4.14 | 2.77 | 0.28 | 0.77 | 0.22 | 0.18 | 2.43 |
| Type I | 21.01 | 4.77 | 2.45 | 63.97 | 3.15 | 2.81 | 0.75 | | | | |
| ASA** | 81.46 | 8.03 | 1.99 | 1.71 | 0.25 | 0.02 | 1.86 | 2.51 | 0.26 | 0.15 | 1.19 |
| Quartzite | 83.98 | 8.51 | 1.53 | $<0.05$ | 0.61 | 0.17 | | | | | 4.08 |
| Limestone | 5.89 | 1.36 | 0.12 | 84.50 | 6.59 | 0.08 | | | | | |

* High alkali-cement; ** Active silica aggregate.
† Loss of ignition.

Portland cements, NPC and HAC respectively, were used to prepare the PCC mixes. The gradation of coarse limestone, quartzite, and AS aggregate meets the ASTM standards designation C33–95. The chemical properties of all aggregate and cement types are shown in Table 1.

## Specimen Preparation and Chloride Contamination

To prepare PCC specimens, aggregates were pre-dried for 24 h at 105°C and cooled to normal temperature before mixing a commercially available Portland cement and tap water using a 0.04 $m^3$ mechanical mixer. The quality control measurements, slump, unit weight, and air content were taken in accordance with ASTM standards designations C 143–95 and C 138–95, respectively. In addition, cylinders, 4 cm in diameter and 8 cm long, were cast to obtain the compression strength at different curing times according to ASTM standards designation C 39–95 (18) (Table 2).

Cylindrical specimens, 15 cm in diameter by 15 cm long with a centered (2.5 cm diameter) brass rod, were used to obtain the dielectric properties using the CTL fixture. Special molds were designed. A screw was attached from inside each mold base to ensure the stability of the brass rod (which it was connected to) during PCC casting. The PCC was cast into three layers; each layer was consolidated using a steel rod (25 blows). After consolidating the third layer, a vibrating table was used to ensure that the consolidation was satisfactory. The surface was finished smooth using a trowel. The molds were then left in a moist curing room at 25°C for 24 h, after which they were demolded and the specimens were placed back in the curing room until the measurements and/or treatment was due. Different mixes were prepared at three w/c ratios, 0.35, 0.45, and 0.55, with three types of aggregate (limestone, quartzite, and AS), with and without air entrainment.

Two methods of chloride contamination were applied. In method I, NaCl was added to the mixing water at different content levels. To achieve the required content of chloride, NaCl was added at the ratio 58/35 of the intended chloride content. Three intended chloride contents (in PCC) were added, 0, 1.25, and 4.76 kg/$m^3$ of PCC. NaCl was dissolved in pure water by continuous mixing using a rod. The contaminated water was then used, as usual, in the PCC mixing process.

TABLE 2
Quality control measurement of PCC mixes.

| Mix* | Slump (cm) | Density (kg/m$^3$) | Strength† (MPa) | Air content (%) |
|---|---|---|---|---|
| A3NS | 12.7 | 2339.1 | 45.4 | 1.5 |
| A4NS | 12.1 | 2322.4 | 36.8 | 1.8 |
| Q3N | 6.0 | 2369.9 | 47.7 | 1.7 |
| Q4N | 7.0 | 2331.4 | 48.6 | 1.4 |
| Q5N | 8.9 | 2291.7 | 32.5 | 1.4 |
| Q3A | 7.4 | 2345.8 | 46.3 | 7.0 |
| L4N | 12.7 | 2416.0 | 54.3 | 1.6 |

* Q, L, and A refer to quartzite, limestone, and active silica aggregate, respectively; 3, 4, and 5: refer to 0.35, 0.45, and 0.55 w/c ratio, respectively; N: refers to non-air entrainment; S refers to using HAC; mixes refered to by three acronyms are made by NPC.

† Compressive strength at 28 days of curing.

In method II, after the PCC specimens were moist-cured for 28 days, they were immersed partially in a NaCl solution (6% by wt). Cylindrical specimens prepared for CTL measurements were placed 3.8 cm in the NaCl solution. Plastic sheets, with holes of the size of the surface areas of immersed specimens, were used to cover the container used for immersing. This process was done to accelerate chloride intrusion into PCC by the aid of the osmotic effect.

The dielectric properties were evaluated over time for specimens treated with methods I and II. Periodically, powdered PCC samples were collected by a drill hammer with a 1.2 cm bit. Six samples were collected from 0 to 2.5 and 2.5 to 5 cm depths located at 2.5 cm, 7.5 cm, and 10 cm from the upper surface of the cylinders. Chloride content was measured, using the Virginia Tech procedure (19). For PCC specimens, contaminated by immersion, the overall chloride content in the PCC was obtained by averaging the measured chloride contents at different depths and distances. Whereas, an average of three measurements was used to identify the chloride content in PCC specimens contaminated by addition of NaCl to the mixing water.

## Dielectric Properties Measurements

Before wave signal measurements, all PCC specimens were removed from the moist curing room and conditioned for 30 min. to 1 hour at room temperature to achieve complete surface drying. For the specimens immersed in NaCl solution, complete surface cleaning of NaCl deposits was done before the specimens were left at room temperature for surface drying. The CTL specimens were placed in the coaxial fixture and the waveform was acquired using the 11801 Digital Sampling Oscilloscope (Fig. 1). The dielectric properties measurements were calculated, according to the methodology illustrated earlier, using Matlab Software.

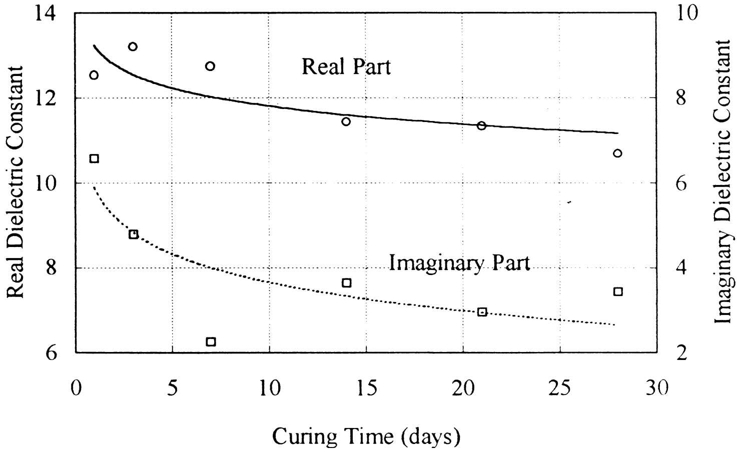


FIG. 2.
Effect of curing time on the dielectric properties (real and imaginary parts) for A3NS specimens (at 550 MHz).

## Discussion

### Basic Properties

The results, in general, showed a great change in the complex dielectric properties within the first 3 days of curing; typical behavior can be seen in Figure 2. After 1 day of curing, the complex dielectric properties showed the highest dielectric properties, which had decreased over curing time. Similar behavior can be seen for different PCC specimens.

In most PCC specimens, the plateau behavior of the complex dielectric properties after one day of curing reflects its low sensitivity to changes in the PCC mixes. This is attributed to the low contribution of the bound water (high percentage at late curing periods) to the overall relaxation of the water, as well as the low sensitivity of PCC conductivity to the change in water content and/or state. The highest percentage of free water and ionic production rate happened to be at one day of curing, which resulted in the highest dielectric properties response. Neither the real nor the imaginary dielectric properties changed with the w/c ratio (Fig. 3). This can be explained by the low sensitivity of both the real and imaginary parts of the dielectric properties to moisture at this frequency (20).

The real dielectric properties of PCC specimens prepared at a 0.45 w/c ratio with limestone (L4N) were higher than that of PCC specimens with quartzite at the same w/c ratio (Q4N) (Fig. 4). A steady difference of almost three in the real dielectric properties over the curing time can be noted. The imaginary dielectric properties is statistically the same for both PCC mixes.

This behavior can be related to the relatively low complex dielectric properties of the PCC and the relatively high dielectric properties of limestone aggregate (eight for limestone and four for quartzite). Thus, the overall PCC dielectric properties were shaped by the aggregate, which made up almost 60% of the total absolute volume of the PCC specimens. This was

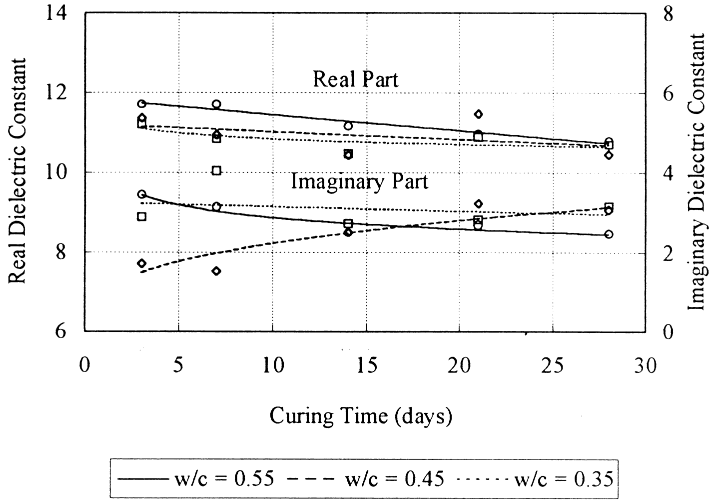


FIG. 3.
Effect of curing time on the dielectric properties (real and imaginary parts) for PCC specimens using quartzite at different w/c ratios (at 550 MHz).

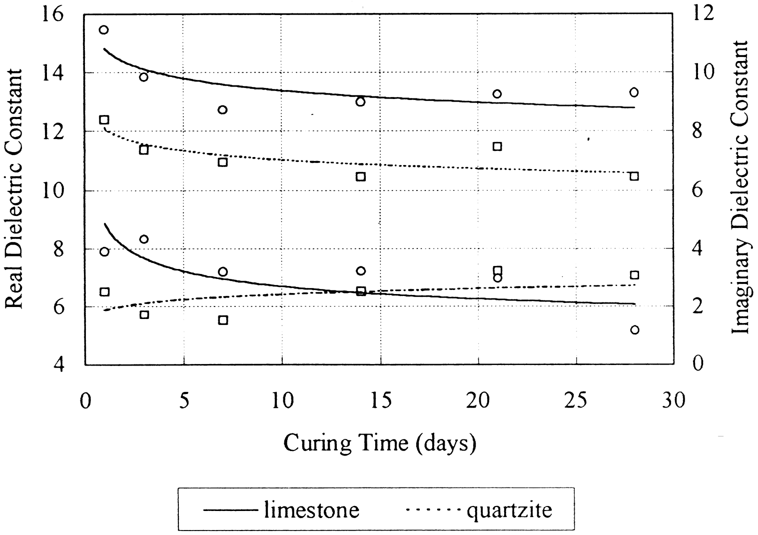


FIG. 4.
Effect of curing time on the dielectric properties (real and imaginary parts) for PCC specimens at a 0.45 w/c ratio using two different types of aggregate (at 550 MHz).

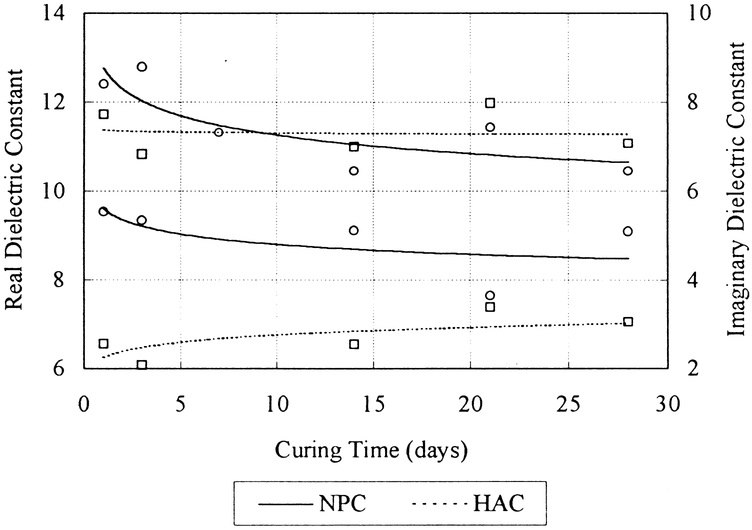


FIG. 5.
Effect of curing time on the dielectric properties (real and imaginary parts) for PCC specimens at a 0.45 w/c ratio using quartzite and two different types of cement (at 550 MHz).

indicated by the steady difference in the real dielectric properties and the insignificant difference in the imaginary dielectric properties over the curing period.

This indicates no contribution of the microstructural changes in the PCC porosity and the pore size distribution to the overall PCC dielectric properties. The structural changes due to the effect of the aggregate transition zone narrowed the difference in the real dielectric properties for both L4N and Q4N specimens at the low RF band (21). The insignificant difference in the imaginary dielectric properties is related to relatively higher conductivity losses of PCC with quartzite, which balances the higher contribution (to the imaginary part) of the real dielectric properties in PCC with limestone.

The real part of the dielectric properties was not affected by the type of cement used in preparing the PCC specimens. The imaginary part of the dielectric properties indicated significant difference between PCC specimens with HAC compared to those NPC (Fig. 5). This behavior suggests that the difference in the water content and/or state between the two different mixes was not sufficient to produce a significant changes in the water polarization. Also, the high imaginary part of the dielectric properties for PCC with HAC reflects the high ion content. The latter explanation agreed with changes observed in the real dielectric properties, at the RF, which is most probably attributed to ion consumption and production (21).

The real and imaginary dielectric properties measurements showed insignificant difference between air entrained and non-entrained PCC specimens (Fig. 6). Since the forms of dominant polarization (which is sensitive to moisture and ion content changes) are absent and/or provided low contribution in this case [20], the influence of air-entrainment was not detected in the PCCs complex dielectric properties.

The response of the real dielectric properties at the microwave frequency depended on both the frequency as well as the maturity of PCC. For most PCC mixes, the real dielectric

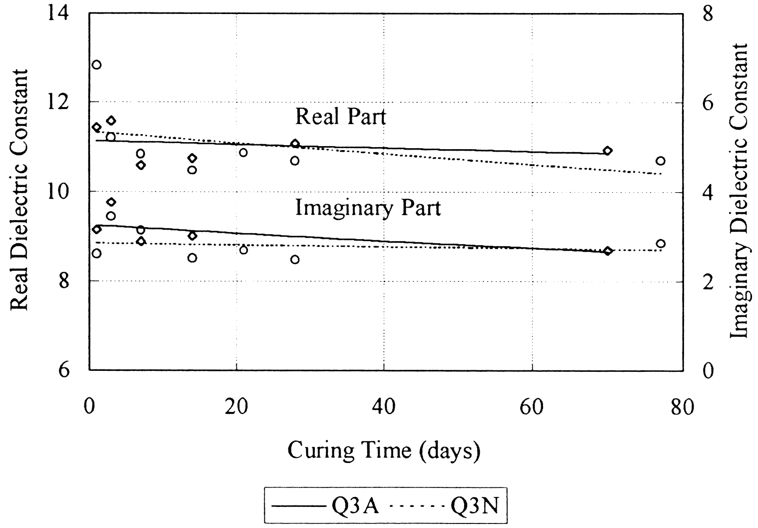


FIG. 6.

Effect of curing time and air entrainment on the dielectric properties (real and imaginary parts) for PCC specimens at a 0.35 w/c ratio using quartzite (at 550 MHz).

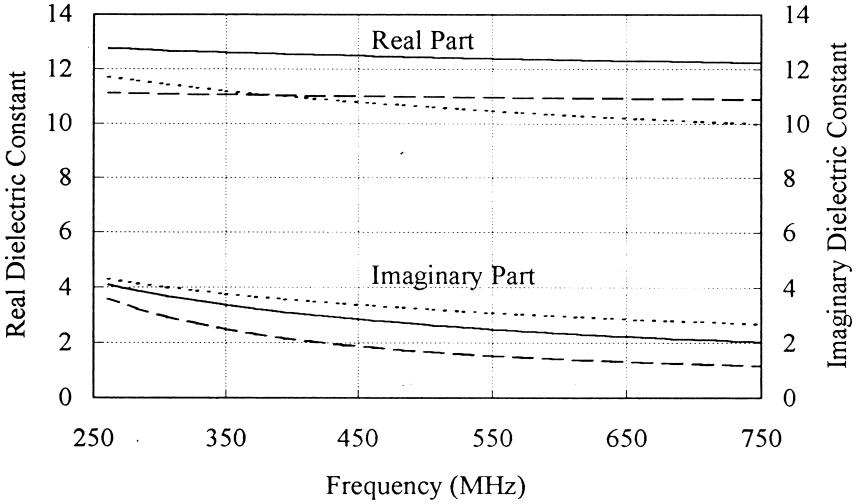


FIG. 7.

Dielectric properties (real and imaginary parts) vs. frequency for Q4N specimens cured at different curing periods.

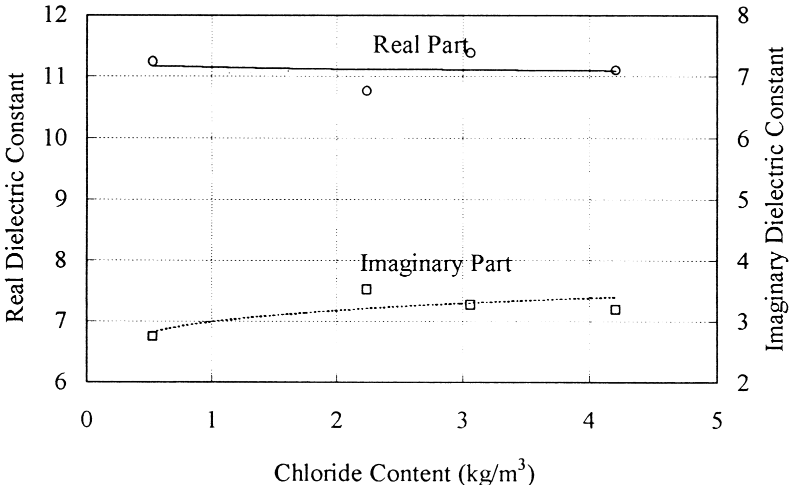


FIG. 8.
Effect of chloride intrusion on the dielectric properties (real and imaginary parts) for Q4N specimens (at 550 MHz).

properties and frequency relationship showed higher decrease rate with further curing (Fig. 7); the imaginary dielectric properties, however, showed a plateau behavior without specific trend.

The behavior of the imaginary dielectric properties, in PCC specimens that showed no specific trend with further curing, implicate low sensitivity of conductivity and relaxation

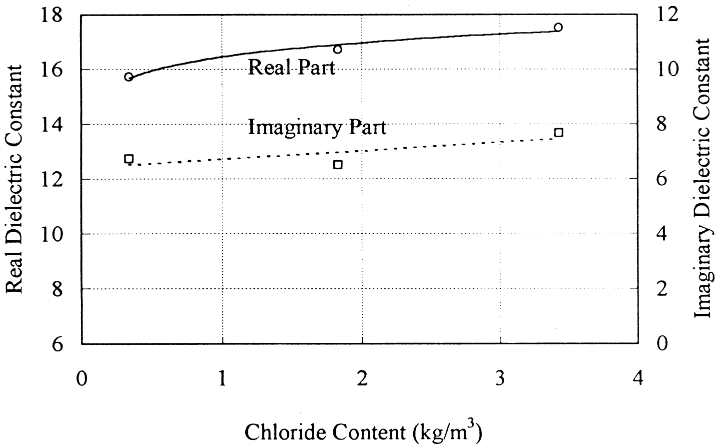


FIG. 9.
Effect of induced chloride on the dielectric properties (real and imaginary parts) for L4N specimens after being cured for 7 days (at 550 MHz).

losses to changes in water state and content due to further hydration. In PCC specimens that showed lower imaginary dielectric properties with curing time, on the other hand, the change in the water (content and state) with curing was significant and produced changes in the conductivity and the relaxation losses. The latter were reflected in the imaginary dielectric properties showing lower values at the same frequencies.

## Chloride Contamination

For PCC specimens contaminated by partial immersion in NaCl solution, the results indicated insignificant changes in the real and imaginary dielectric properties with the amount of intruded chloride (in kg/$m^3$ of PCC) (Fig. 8). The significance of the change was based on a coefficient of variation (COV) of 5 to 10% for the real and imaginary parts of the dielectric properties.

For PCC specimens contaminated by the addition of NaCl to the mixing water, the results do not indicate strong evidence for changes in the complex dielectric properties with chloride after being cured for 7 days (Fig. 9). Similar behavior can be noted for same specimens, but cured for three and 40 days. The insignificant change in the real and imaginary dielectric propertiess for the PCC specimens apparently refers to the low effect of $Na^+$ and $Cl^-$ ions on the relaxation time and conductivity losses of the capillary water, respectively (22). The previous conclusions were built based on a COV of 5% to 10% for the real and the imaginary dielectric properties.

## Conclusions

Based on the previous discussion, the following can be stated

1. The dielectric properties of PCC decreased over curing time.
2. The aggregate type has significantly affected PCC dielectric properties.
3. The dielectric properties of PCC specimens showed low sensitivity to change in cement type or air entrainment.
4. The chloride presence did not affect significantly the dielectric properties of PCC specimens.

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