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# Microwave Reflection and Dielectric Properties of Mortar Subjected to Compression Force and Cyclically Exposed to Water and Sodium Chloride Solution

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*Abstract*—Corrosion of the reinforcing steel is a major cause of damage and deterioration in reinforced concrete structures such as concrete bridge decks and columns. Chloride intrusion into concrete can lead to depassivation of the steel and initiation of corrosion. Thus, it is very important to be able to nondestructively detect and evaluate the free chloride content in concrete. Near-field microwave nondestructive testing techniques, using open-ended rectangular waveguide probes, have shown great potential for evaluating various properties of concrete, including the successful detection of sodium chloride added to mortar mixing water. In this study, several mortar samples are cyclically soaked in distilled and salt water while also experiencing compression force. Compression force, simulating in-service loading, causes microcracking, which results in increased microcracking and permeability, promoting chloride ingress. The daily microwave reflection and dielectric properties of these samples were measured at 3 GHz. The results show the capability of these microwave measurements for detecting the increased level of chloride permeation and loading as a function of the increasing number of soaking cycles. The influence of salt ingress is shown to be more prominent in the loss factor, while the effect of loading is more evident in the permittivity of the samples.

*Index Terms*—Chloride, concrete, microwaves, nondestructive testing, reinforcing steel.

## I. INTRODUCTION

ONE OF THE primary causes of damage and deterioration in reinforced concrete structures is the corrosion of the reinforcing steel [1]. When a steel reinforcing bar corrodes, the loss of load-bearing steel and the generation of stresses by the formation of corrosion products, which occupy a larger volume than the original material, may lead to the deterioration and eventual failure of the reinforced concrete structure. Reinforcing steel corrosion is the cause of damage in the majority of reinforced concrete bridges in the United States [2]. As has been experienced in the past few decades, vulnerability and eventual deterioration of critical structures to reinforcement corrosion have been on the rise. This has been due to in-service factors, such as the use of deicing salts, exposure of structures to salt water environments, as well as design procedures that emphasize compressive strength instead of long-term durability.

Normally, the high pH level in concrete causes a passive film to form on the reinforcing steel, protecting it from corrosion. However, this film is destroyed when a sufficient concentration of chloride ions is present at the steel/concrete interface. Subsequently, through an electrochemical process in the presence of oxygen and moisture, the corrosion of reinforcing steel takes place. The amount of free chloride ions in the pore solution is the important factor used as the limit set for the amount of chloride in reinforced concrete [3].

Currently, there are two standard tests used to measure the penetration of chloride into concrete. These tests, which are commonly referred to as the "rapid chloride permeability test," determine the total electrical charge passed during a certain time interval through a concrete disk between solutions of sodium chloride and sodium hydroxide when a potential difference of 60 V dc is maintained [4], [5]. The charge is related to the penetrability of the concrete to chloride ions, which can be of help when comparing concrete mixes. These tests do not closely replicate the transport of chloride ions in field conditions. Another test for chloride content is commonly referred to as the "90-day ponding test" [6]. This test subjects a concrete sample to a chloride solution over a given amount of time. It is generally regarded as an appropriate test, but the test period can be lengthy. After a sufficient period of chloride exposure, the sample is ground, and the amount of penetrated chloride is measured. Results from this test take at least 90 days to produce, but with the newer high-strength mixes, the results may take longer due to the slow ingress of chloride from this test.

Development of a reliable test method for in-situ measurement of the chloride penetration in in-service concrete would be invaluable to the concrete and construction industry. From an economic standpoint, accurate measurements of the concentration of chlorides and penetration depth in concrete structures would allow agencies to more effectively allocate funding for repair of those structures with the most critical needs.

## II. BACKGROUND

Microwave nondestructive testing and evaluation (NDT&E) techniques, mostly in the near-field using open-ended rectan-

gular waveguide probes and monopole antennas, have been used to interrogate a wide variety of cement-based materials for their important physical and structural properties, namely

- evaluation of water-to-cement ratio ($w/c$) and compressive strength of hardened cement paste [7], [8];
- evaluation of fresh concrete $w/c$ [9];
- evaluation of porosity and sand-to-cement ratio ($s/c$) in mortar [10], [11];
- evaluating mortar permittivity using a combined microwave near-field and modulated scattering technique [12];
- evaluation of coarse aggregate-to-cement ratio ($ca/c$), cure-state and material properties of concrete [13]–[16];
- detection of grout in masonry bricks [17];
- detection of delamination between hardened cement paste and fiber-reinforced polymer (FRP) composites [18], [19].

In recent years, near-field microwave NDT techniques have also been extensively used in evaluating chloride contamination in cement-based materials. It has been shown that the addition of salt to the mixing water of mortar can be easily evaluated using these techniques [20]. It has also been shown that the influence of cyclical chloride ingress in mortar exposed to chloride solution can also be evaluated using these techniques [21]–[25].

In practice, many of the cement-based structures that are exposed to chloride solution, such as bridge decks, also experience cyclical loading (i.e., caused by traffic). Under sufficient loading, pre-existing microcracks can grow, and new cracks may form. This can result in higher permeability and hence increased chloride penetration in the structure. To this end, an investigation was undertaken in which several mortar cubes were cyclically exposed to a combination of distilled water, sodium chloride solution (salt water), and compression stress. In this way, the influence of sodium chloride ingress as well as compression stress could be, potentially and separately, evaluated. The experimental approach and the results of this investigation are described in this paper.

## III. APPROACH

Five 8 in $\times$ 8 in $\times$ 8 in $\times$ (200 mm $\times$ 200 mm $\times$ 200 mm) mortar cubes all with $w/c$ of 0.50 and $s/c$ of 2.5 were produced using tap water and Portland cement type I/II. These cubes were then left in the hydration room for 24 h and subsequently left to cure at room temperature and low humidity for approximately seven months. The cubes were then exposed to distilled water (hereafter referred to as water), sodium chloride solution, and compression force in the following manner.

- One cube was left alone and was used as the reference test specimen, for monitoring the influence (if any) of the ambient conditions and for comparison with the other cubes.
- One cube was cyclically soaked in water.
- One cube was loaded before being cyclically soaked in water.
- One cube was cyclically soaked in a salt-water solution (using ACS-certified NaCl) with 2.8% salinity.
- One cube was loaded before being cyclically soaked in a similar salt-water solution.
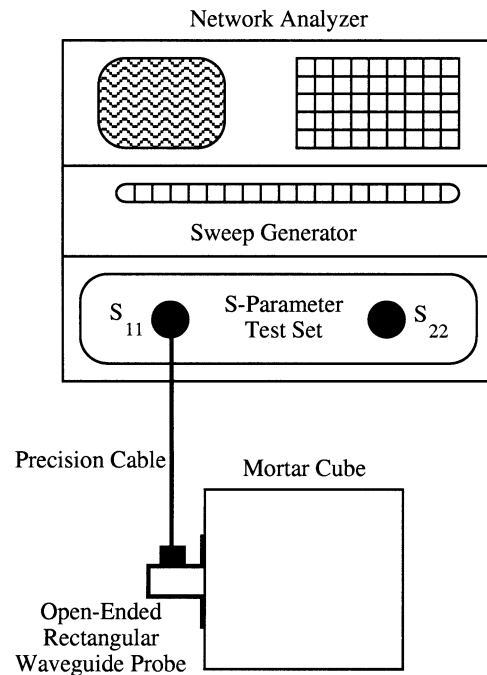


Fig. 1.   Schematic of the measurement apparatus.

The cubes were submerged in their respective solutions to within 1/4 in of their top surface. After the first soaking cycle, one of the cubes soaked in water and one soaked in salt water were loaded to 65 000 lbs. ($\sim$1,015 psi) before each subsequent soaking cycle. This force corresponds to approximately one-third of the average compressive strength of the cubes. It was assumed that one-third of the compressive strength would be sufficient to generate a reasonable and measurable amount of microcracking in the cubes. After 24 h of soaking, the cubes were removed from the solutions and left in ambient conditions for another 24 h. This allowed the excess liquid surface water to evaporate before commencing the microwave reflection property measurements. Microwave measurements on the loaded water-soaked cube were discontinued after cycle 3 due to the presence of significant cracking that had developed on the surface of the cube. This was due to the fact that the surface on which the load was applied was nonuniform, thereby resulting in nonuniform distribution of the load throughout the cube and hence resulting in some large surface cracks.

Subsequently, the daily calibrated near-field microwave reflection coefficients, $\Gamma$ (with a magnitude, $|\Gamma|$ and a phase, $\phi$, of these cubes were measured at S-band (2.6–3.95 GHz) using open-ended rectangular waveguide probes in conjunction with an HP8510 vector network analyzer, as shown in Fig. 1. The cubes represented an infinite half-space of a material at this frequency even before being soaked, as determined in many previous investigations [13], [14], [26]. Each measurement cycle lasted on average approximately 33 days, after which no appreciable changes in the reflection properties were measured. At this time a new soaking and measurement cycle was initiated. The entire investigation included six such cycles. At S-band 16 measurements were conducted on each cube (four per side), in order to obtain an average value of the reflection coefficient.
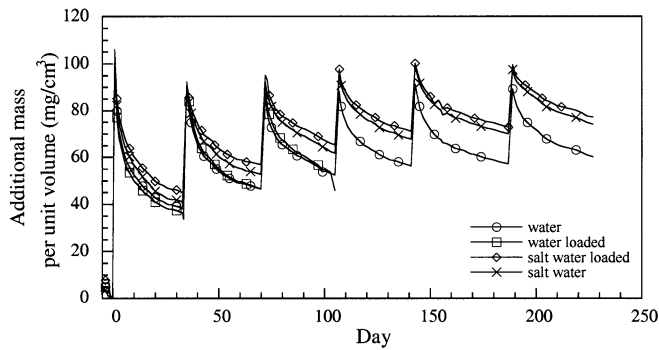
Fig. 2. Daily change in the relative additional mass per unit volume of the cubes.

| Salinity (%) | (Relative Permittivity) – j(Relative Loss Factor) |
|:---:|:---:|
| 0 | 77.34 – j11.98 |
| 2.8 | 69.15 – j35.80 |
| 4.9 | 63.40 – j51.13 |
| 6.0 | 60.55 – j58.87 |

The results reported in the following sections are those at a frequency of 3 GHz, representing typical results at S-band.

The macroscopic behavior of dielectric materials is described by a parameter known as the dielectric constant (dielectric properties). In general, this is a complex parameter whose real part (absolute permittivity) indicates the ability of the material to be polarized or store microwave energy, while its imaginary part (absolute loss factor) indicates the ability of the material to absorb microwave energy. When referenced to the permittivity of free space, these two parameters are called the relative (to free space) permittivity and loss factor of the material, respectively. The dielectric properties of the cubes were also calculated from the measured $\Gamma$ using a previously developed full-wave electromagnetic formulation [26].

## IV. INFLUENCE OF SALT

The mortar cubes are made of cement paste, sand and pores, which may be completely filled with water or air or partially filled with both water and air. When soaked, water or salt solution penetrates into the pores through a capillary draw. Once removed from the solution, a portion of the water continually evaporates from the near surface regions and pores (i.e., first few centimeters). The remaining portion continues to be drawn into the sample core by the capillary draw. Therefore, unless the cubes are slowly oven dried after each soaking cycle, some liquid will remain in the pores. The relative additional mass per unit volume of the cubes was also measured daily prior to each microwave measurement, as shown in Fig. 2. This indicates the gain in mass per unit volume for each cube relative to its mass on day zero (pre-soaked mass). The results clearly show an increase in the relative additional mass per unit volume of all cubes at the end of each cycle with respect to the end of the previous cycle. The results also show the fact that the salt water-soaked cubes gained more in mass per unit volume as a function of increasing number of cycles. This is attributed to two facts, namely, a) after each soaking cycle more salt is deposited into the pores, and b) the addition of salt in the pores slows the evaporation rate of the liquid water from the cubes. In other words, the salt in the pores holds onto the liquid water longer than air-filled pores. The effects of these phenomena are illustrated in Fig. 2 since during the first few days after being removed from their respective solutions, the decrease in additional mass per unit volume is primarily due to liquid water evaporation. The evaporation rate

continually decreases, as a function of increasing number of cycles, for the salt water-soaked cubes, as is evident in Fig. 2 (and, to a much lesser extent, for the water-soaked cube).

As the liquid water evaporates from the pores, it leaves air behind in the water-soaked cubes and free salt crystals (and some air) in the completely dried pores of the salt water-soaked cubes. NaCl is a low-loss dielectric material whose relative permittivity is ~3.5. Some of the salt becomes bound as sodium chloride ions react with calcium aluminate present in the cement paste to form Friedel's salt. The influence, on the microwave reflection properties of the cubes, of free salts in the pores is expected to be more significant than the Friedel's salt, since the latter has a smaller volume content and its dielectric properties are expected to be more similar to cement paste [21]–[23]. Also, as the evaporation process in the salt water-soaked cubes continues, the salinity of the remaining solution increases. At 3 GHz, the dielectric properties of salt water are markedly different than distilled water [27]. The relative permittivity of distilled water is only slightly higher (by percentage) than that of salt water with 2.8% salinity. However, the relative loss factor of salt water with 2.8% salinity is significantly higher than that of distilled water. These differences get larger as the salinity of water increases (i.e., as more pore liquid evaporates). Table I shows the relative permittivity and loss factor for water with four different salinity cases at a frequency of 3 GHz, calculated using the expressions given in reference [27]. Thus, the presence of salt influences the dielectric properties of the cubes, whether the salt is in the solution form or free (in that order of importance).

## V. RESULTS

Fig. 3(a) and (b) show the daily measured $|\Gamma|$ and $\phi$ for the five mortar cubes for the first cycle, respectively. From the measurements at day zero (the day prior to soaking), it is evident that all of the cubes possessed similar reflection properties, as they should since they were all made from the same batch of mortar mix. However, once the cubes are soaked, their reflection properties are expected to change significantly as a result of absorbing water or salt water. This aspect is also confirmed by the fact that from day two on, there is a significant difference in the reflection properties of the reference cube compared to those of the soaked cubes. Once a cube is taken out of the tap or the salt water, its daily measured dielectric properties progressively change, indicating the effect of evaporation of liquid water from, and the capillary draw of water or salt water to-
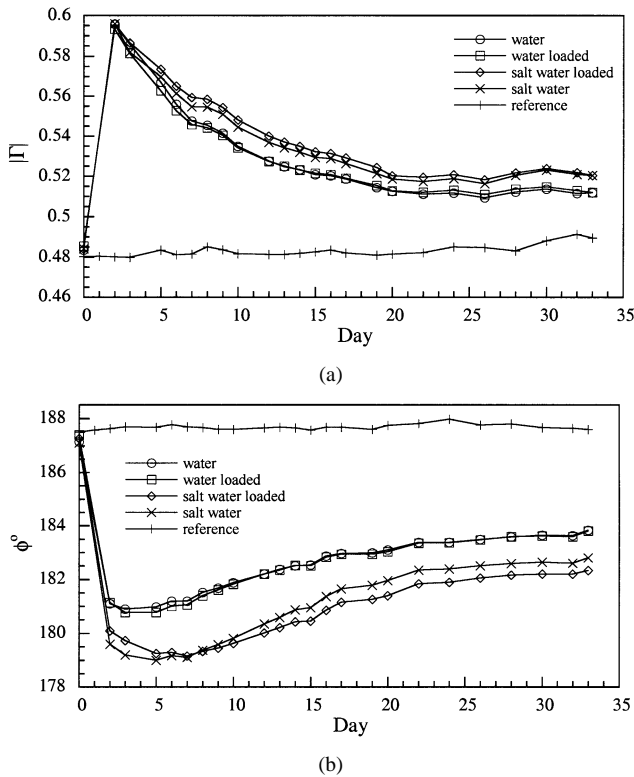
Fig. 3.    Reflection coefficient of the cubes at 3 GHz for cycle 1; (a) magnitude and (b) phase.



Fig. 4.    Dielectric properties of the cubes at 3 GHz for cycle 1; (a) relative permittivity and (b) relative loss factor.

ward the core of the cube. Since prior to the second cycle the cubes had not been loaded, the pair of cubes that were soaked in the water are expected to exhibit similar reflection properties, and similarly so should the cubes soaked in the salt water. This fact is also evident in Fig. 3. However, at 3 GHz, the dielectric properties of salt water are substantially different than distilled water, as mentioned earlier [27]. Consequently, the daily magnitude and phase of the reflection coefficient of the cubes soaked in salt water should also be different than those soaked in water, as also indicated in Fig. 3. During the first few days, this difference is more a function of the difference between the ionic concentration of the pore solutions in the two samples. Toward the final days, this difference is caused by the presence of salt crystals in the near surface pores (i.e., the first few centimeters) and the salinity difference in the solutions left in the pores (more toward the core). To the extent that the microwave signal penetrates into the cube, the former is expected to be more of a significant factor than the latter during the final days of a cycle, since more and more of the cube becomes devoid of the sodium chloride solution as a function of drying time.

The change in the reflection properties of these cubes (as shown in Fig. 3) is also manifested in their respective calculated dielectric properties. Fig. 4(a) and (b) show the calculated daily relative permittivity and loss factor of the cubes, respectively [26]. The results show a similar trend between $|\Gamma|$ and permittivity, and $\phi$ and loss factor, respectively. It must be noted that the results shown in Fig. 4 represent permittivity and loss factor for a homogeneous infinite half-space of a material having a $\Gamma$ equal to that measured from a cube. This is important since complete homogeneity is only achieved during the first couple
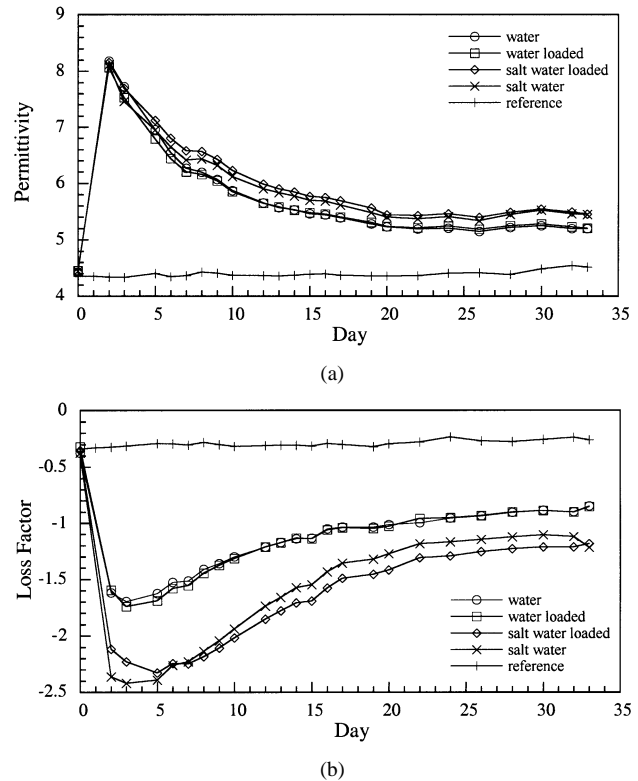
of days or so after removal of the cubes from the solutions and after approximately day 20, as shown in Fig. 4. In between, the evaporation of liquid water is not uniform, producing a gradient of water distribution in the cube. Thus, the values of permittivity and loss factor shown in Fig. 4 should be considered with this fact in mind. However, ultimately, one is interested in the dielectric properties of these cubes after many days of having been taken out of their respective solutions, since, at this time, changes in the dielectric properties are indicative of the fact that salt crystals have been deposited in the pores. At this time, each cube can be considered a homogeneous half-space of a material, and the calculated dielectric properties indicate its average dielectric properties.

The results are shown in Fig. 4(a) and (b) for two regions; namely, the first few days and the last few days must be studied in more detail. As mentioned earlier, at 3 GHz, the permittivity of distilled water is slightly higher than the permittivity of salt water, while the loss factor of salt water is substantially higher than that of distilled water (see Table I). Thus, during the first few days of the cycle, the permittivities of the water-soaked and the salt water-soaked cubes are nearly the same (within the measurement error). However, the salt water-soaked cubes exhibit a slightly higher permittivity after the first few days. As discussed earlier, this is attributed to two factors. First, the salt reduces the evaporation rate of the liquid water, and thus the two salt water cubes hold moisture for a bit longer than the water cubes, rendering higher permittivities for the former cubes. Second, as the salt water evaporates, it replaces the air in the pores with salt crystals, which also results in an increase in the average permittivity of these cubes (some salt water may also remain in the
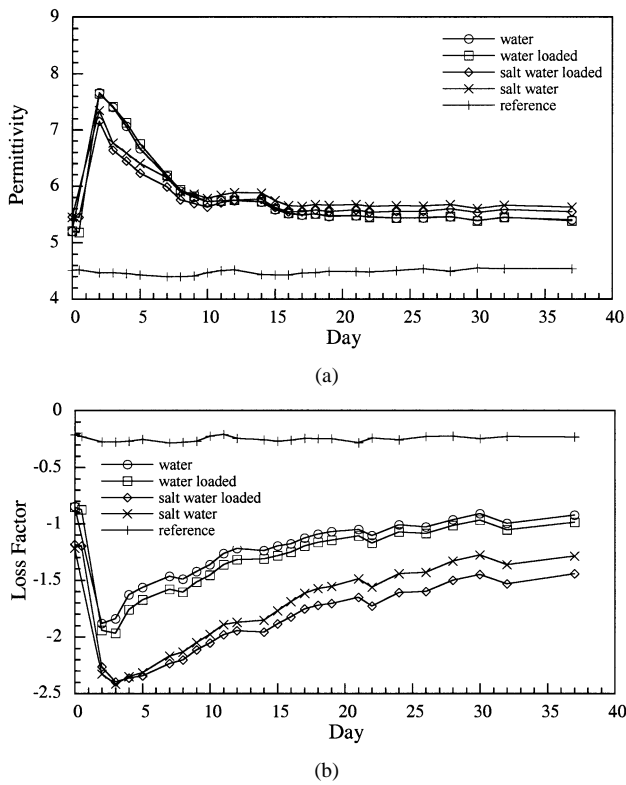
Fig. 5.   Dielectric properties of the cubes at 3 GHz for cycle 2; (a) relative permittivity and (b) relative loss factor.



Fig. 6.   Dielectric properties of the cubes at 3 GHz for all cycles; (a) relative permittivity and (b) relative loss factor.

inner pores). This fact is best shown during the final days of the cycle, as most of the liquid water has evaporated from the near surface regions of the cubes and some free salt has been deposited in the pores of the salt water-soaked cubes.

The influence of salt water on the loss factor is more pronounced [Fig. 4(b)], as expected. During the first few days, the loss factor of the salt water-soaked cubes is substantially higher than the water-soaked cubes, with the same being true (to a lesser extent) during the last days of the cycle since much of the liquid water has evaporated by this time (loss factor is commonly shown as a negative value). The results of cycle 1 clearly show the influence of salt water in these cubes.

Fig. 5(a) and (b) show the permittivity and loss factor for cycle 2, respectively. When a mortar cube is soaked, it absorbs free water by means of capillary action. The presence of microcracks aids in the absorption process by increasing the amount of liquid water trapped in the pores, thereby affecting the dielectric properties of the cube. In the case of salt water-soaked cubes, more salt water is drawn into the pores. Subsequently, as the liquid water evaporates, it leaves behind salt crystals in the pores. Thus, the effect of loading is expected to affect the dielectric properties of the loaded salt water-soaked cube more than the loaded water-soaked cube. This is clearly indicated in the permittivity and loss factor results for cycle 2, as compared to the same results for cycle 1 (in particular for the loss factor). It is also important to note that the permittivity of the water-soaked cubes during the first few days is higher than the salt water-soaked cubes, as expected. However, the loss factor results for the salt water-soaked cubes show that the loaded cube absorbed more salt solution, due to the increased permeability.
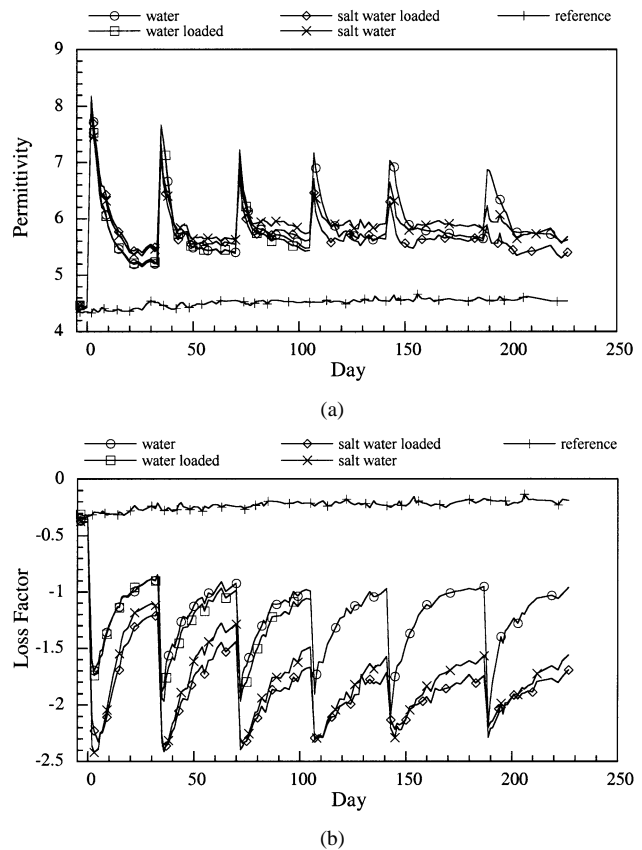
This is a clear indication of the influence of loading. The same is also true, but to a lesser extent, for the water-soaked cube.

Fig. 6(a) and (b) show the permittivity and loss factor for all six cycles. During the first few days of each cycle the water-soaked cube shows higher permittivity than the salt water-soaked cube, and this trend becomes more prominent as the number of cycles increases. As explained earlier, this is due to the fact that the permittivity of water is higher than salt water at this frequency. It is also interesting to note that the loaded salt water-soaked cubes present the lowest permittivity during the final three cycles, since the amount of salt water in the pores of this cube progressively increases, as also expected.

As the number of cycles increases, several interesting phenomena occur regarding the loss factor results [Fig. 6(b)]. First, the difference between the salt water-soaked loaded and not loaded cubes increases toward the end of each cycle, which indicates the overall effect of increased permeability. Second, for the water-soaked cubes, the loss factor at day one of each cycle increases as a function of increasing number of cycles, while for the salt water-soaked cubes, the loss factor remains fairly constant (within the measurement error). For these cubes, as the number of cycles increases, more salt is deposited in the pores. The salt concentration tends to want to reach an equilibrium between that found in the pores and the surrounding solution. The concentration in the pores upon rewetting is expected to be quite high; hence, the driving force for additional salt ingress may be lower with subsequent exposure. For the water-soaked cubes, at the conclusion of each cycle more liquid water evaporates

from its pores, and more liquid water is soaked in during the next cycle compared to the salt water-soaked cubes. Leaching may also cause a slight increase in the very near surface pores, which during the early days of each cycle are filled with water, and hence may cause a slight increase in the loss factor. At the last day of each cycle and for the water-soaked cube, most of the liquid water has evaporated from the near surface pores, and, hence, the loss factor at the end of the cycles remains fairly constant. For all cycles, the daily rate of change in the loss factor remains fairly unchanged for the water-soaked cubes. However, the same slope substantially decreases for the salt water-soaked cubes. This phenomenon is thought to be due to the presence of increasing free salt content in these cubes as a function of increasing number of cycles. As more and more salt is left behind, more liquid water is captured by the salt, which reduces the evaporation rate.

As explained earlier, the results obtained during the last days of each cycle are important for determining the potential influence of free salt content in the cubes. Fig.7 (a) and (b) show the permittivity and loss factor for day 33 of each cycle, respectively. The permittivity for the water-soaked cubes increases as a function of increasing number of cycles. As explained earlier, this is due to the fact that unless the cubes are slowly oven dried after each soaking cycle, water remains in the cubes more concentrated toward the core (e.g., water movement toward the core due to the capillary draw). The salt water-soaked cubes show a decreasing trend in permittivity after the fourth cycle. This is attributed to the fact that the salt left behind in the remaining liquid solution causes a reduction in its permittivity, and this trend becomes more prominent the more the salinity of the pore solution increases. It is also important to note that the loaded salt water-soaked cube, with a resulting increase in permeability, absorbs more salt solution, and hence its permittivity decreases more as a function of increasing number of cycles. This is clearly an indication of the influence of loading.

An examination of Fig. 7(b) shows that the loss factor for the salt water-soaked cube is larger than the water-soaked cube, as expected (more free salt and salt water in the pores). The loss factor for the salt water-soaked cubes continues to increase as a function of increasing number of cycles, as well. However, it is important to note that the loss factor is consistently larger for the loaded cube, which is a direct result of having more pore volume filled with more salt water and/or salt crystals. These results collectively show the influence of salt permeation and loading on the cubes. Thus, a comparison of Fig. 7(a) and (b) indicates that the influence of salt is more prominent in the loss factor, while the effect of loading is more evident in the permittivity of the cubes.

## VI. CONCLUSION

Nondestructive detection and evaluation of sodium chloride ingress in cement-based materials and structures are important issues in the construction industry. Near-field microwave nondestructive testing and evaluation techniques have shown great promise for the inspection of a wide range of properties associated with these materials. In this investigation, several
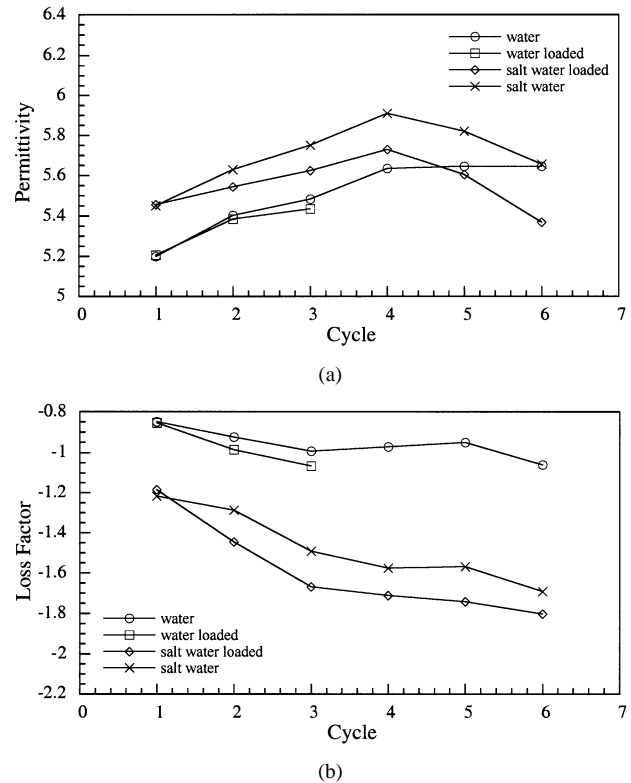


Fig. 7. Dielectric properties of the cubes at day 33 of the six cycles; (a) relative permittivity and (b) relative loss factor.

mortar cubes were cyclically soaked in distilled water and salt water having a salinity of 2.8%. Compression stress, equal to approximately one-third of their compressive strength, was also applied to some of the cubes before each soaking cycle to simulate the in-service loading that such materials may experience. Microwave reflection and dielectric property measurements of these cubes, at a frequency of 3 GHz, showed the capability of detecting the presence of salt and salt water in these mortar cubes. Some factors, such as the inability of all of the liquid to evaporate from the cubes during each cycle, were also demonstrated. Moreover, the influence of the compression stress, which is expected to increase the permeability, was also detected. The results indicated that, at the end of each cycle, the influence of salt is more prominent in the loss factor, while the effect of loading is more evident in the permittivity of the cubes.

Further investigation in developing an electromagnetic model, describing these phenomena, is currently ongoing. It is expected that such a model can be used to give the actual salt content in such cubes as a function of soaking cycle and compression force. Ground truth or physical data, such as the daily additional mass of the cubes, will also be a useful and complementary piece of information in the development of this model and will lead to a better understanding of the salt ingress in these cubes. Such data can be obtained by means of chemical analysis. This issue is also being considered for future work in this area. Overall, the results are very encouraging, since they are nondestructive and have yielded valuable information about the difference caused by the salt water and salt content in these cubes.

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