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Effect of Exposure Conditions on the Long-Term Dielectric Properties of Mortar Samples Containing ASR Gel

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Abstract. Alkali-silica reaction (ASR) is a chemical reaction between alkalis present in portland cement and amorphous or otherwise disordered siliceous minerals in particular aggregates. Through this reaction, reactive silica binds with hydroxyl and alkali ions and forms a gel, known as ASR gel. Recently, microwave materials characterization techniques have shown great potential for detecting ASR in mortar. However, the comprehensive understanding of variables that affect the extent of ASR in mortar and their interaction with microwave signals, in particular the effect of environmental exposure conditions requires more investigations. Therefore, parameters related to these conditions must be considered when using microwave techniques for ASR detection and evaluation. In this paper, the effect of exposure conditions on ASR gel formation and microwave dielectric properties of mortar samples is investigated. To this end, extended measurements of the complex dielectric constants of three different sets of mortar samples are presented at S-band (2.6 – 3.95 GHz). The samples were cast with potentially reactive ASR-aggregates and subjected to different environmental conditions. The results show slightly different permittivities for the differently stored samples, potentially indicating different amount of ASR gel. This observation was corroborated through UV fluorescence microscopy, where different amounts of ASR gel were observed in the samples. Moreover, the results indicate that ASR gel evolution may be better tracked through loss factor measurements, while pre-existing-gel may be better detected through permittivity measurements.

INTRODUCTION

Alkali-silica reaction (ASR) is a chemical reaction between alkalis present in portland cement and amorphous or otherwise disordered siliceous minerals in particular aggregates (i.e., ASR-reactive aggregates). In this reaction, reactive silica binds with hydroxyl and alkali ions and forms an alkali-silica reaction gel, known as ASR gel [1]. ASR gel may imbibe water (moisture) from its surroundings and expand. If the tensile stress caused by the expansion of gel exceeds that the tensile capacity of aggregate/paste, it creates microcracking within aggregate, paste, and at their interface. As long as sufficient moisture (typically internal RH > 80%) is available, the reaction progresses and may result in the higher extent of microcracking and deterioration of concrete structures. There are three main requirements for ASR gel formation, namely: sufficient alkali, amorphous or strained (disordered) silica, and moisture. To bring a better understanding to ASR gel formation from dielectric property measurements standpoint, one must monitor the influence of environmental conditions as it affects the availability of moisture in cement-based materials.

Microwave materials characterization techniques have shown the capability of evaluating a number of critical properties associated with cement-based materials, such as: material content [2], chloride permeation [3], [4], carbonation [5], and most recently ASR gel formation [6]–[10]. Microwave signals are sensitive to the presence of moisture within dielectric materials. In general, the interaction of microwave signals with dielectric materials is described by a parameter known as the complex dielectric constant, which is intrinsic to the material, and is independent of the method with which it is measured. This complex parameter is referred to as relative dielectric constant (ε_r) once it is referenced to the dielectric constant of the free-space. As indicated in (1), the complex-valued relative dielectric constant can be further defined by its real and imaginary parts, where the former (relative

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permittivity) indicates the ability of the material to store microwave energy and the latter (relative loss factor) indicates the ability of the material to absorb microwave energy:

$$\varepsilon_r = \varepsilon'_r - j\varepsilon''_r \quad (1)$$

The complex dielectric constant of mortar is affected by environmental conditions, as these influence not only the rate of ASR but also the reaction of cement. In fact, particularly at early ages, the duration and type of curing of mortar samples affects the rate and extent of cement hydration—a chemical reaction between water and cement grains in which part of free water transforms to the chemically bound water of reaction products—and the dielectric constants of hydration products. Given that mortar cures over time, monitoring of its temporal complex dielectric constant can provide information about its curing process as well as ASR formation in the presence of ASR-reactive aggregates.

This paper presents long-term measurements of relative dielectric constants of three different sets of mortar samples at S-band (2.6 – 3.95 GHz), cast with potentially ASR-reactive aggregates, and stored at different conditions. Mortar samples were exposed to both humid and dry conditions for two reasons. First, to determine the potential relationship between different storage conditions and the ultimate (final) dielectric constant of the samples. Second, to investigate whether any difference in the ultimate dielectric constants of the mortar samples correspond to the amount of produced ASR gel. The first objective is achieved by monitoring the long-term dielectric constant of the samples, stored at different environmental conditions. The second objective is accomplished by examining the correlation between the amount of ASR gel formation and the dielectric measurements of the samples, through UV fluorescence microscopy images of the samples.

SAMPLE PREPARATION AND CURING CONDITIONS

Three sets of mortars, each set having two similar samples were cast with an aggregate-to-cement (*a/c*) ratio of 2.25 and water-to-cement (*w/c*) ratio of 0.47. Furthermore, the reactivity of the aggregate type was examined following the ASTM C1260. The standard classifies the aggregate type as potentially reactive since the average fourteen-day expansion of mortar bars cast with this aggregate type was 0.383%, which exceeds the fourteen-day expansion limit of the ASTM C 1260 standard. To accelerate ASR gel formation, sodium hydroxide (NaOH) was added to the mixing water to achieve 1.25% soda equivalent by mass. Each sample was cast in a polymethylmethacrylate mold with a cross section of 7.21 cm × 3.4 cm, corresponding to the S-band rectangular waveguide cross-section dimensions. The length of the samples were ~2-3 cm (the dielectric constant measurements are independent of sample length). Every sample within each set was exposed to different temperature and relative humidity (RH) levels for different time periods. Hot and humid conditions (39ºc ± 1ºc and 91% ± 5% RH) was realized by keeping samples above water in a sealed container in an environmental chamber similar to the conditions for the prism test [11]. Ambient condition was measured to be (24ºC ± 1ºC and 35% ± 10% RH).

Samples in batch #1 were cured in the chamber for 28 days, and remained there for an additional 152 days. After 180 days, sample 1 was removed from the chamber and put in ambient conditions, while sample 2 remained in the chamber. On day 320, sample 2 was also removed from the chamber and put in ambient conditions until the end of the experiment.

Samples in batch #2 were cured for the initial 28 days in the chamber. Afterwards, sample 3 was removed from the chamber and kept in ambient conditions for the rest of the experiment. Sample 4 was also removed from the chamber after the first 28 days. However, it was returned into the chamber on day 180, for 140 days. Afterwards it was put back in ambient conditions.

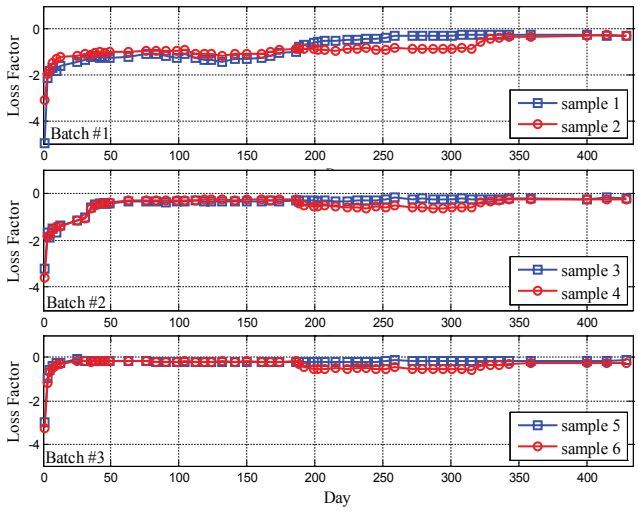
Contrary to the batch #1 and batch #2, samples in batch #3 were kept in the ambient conditions during their first 28 days of curing. However, after 180 days, sample 6 was put in the chamber for 140 days, and then returned back to ambient conditions. Sample 5 was out of the chamber for the entire period of the measurements. Table 1, summarizes the storage conditions of the samples within each set.

TABLE 1. Conditioning of the samples.

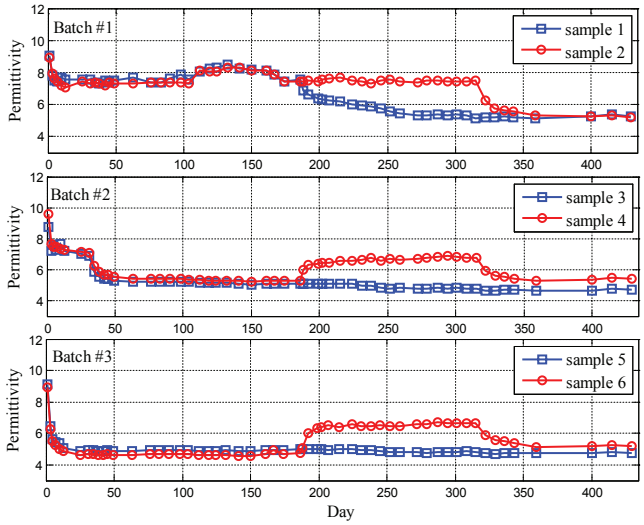
Mortars		Day 1-28	Day 28-180	Day 180-320	Day 320-430
Batch #1	**Sample 1**	Chamber	Chamber	Ambient	Ambient
	Sample 2	Chamber	Chamber	Chamber	Ambient
Batch #2	**Sample 3**	Chamber	Ambient	Ambient	Ambient
	Sample 4	Chamber	Ambient	Chamber	Ambient
Batch #3	**Sample 5**	Ambient	Ambient	Ambient	Ambient
	Sample 6	Ambient	Ambient	Chamber	Ambient

DIELECTRIC PROPERTY MEASUREMENT RESULTS

The relative permittivity and loss factor of each sample were measured on a weekly basis using the completely-filled waveguide technique outlined in [12]. The measured relative permittivity and loss factor of the samples are shown in Figs. 1a and 1b, respectively. As it can be seen during the time that samples were kept in the chamber a higher (relative) permittivity and loss factor (loss factor is a negative-valued number) were measured for the samples, and during the exposure to ambient conditions those values started to decrease. These trends were expected, since additional water was introduced to the samples during humid conditions, this additional water increased the dielectric constant of the samples. On the other hand, during the drying period, evaporation of water caused the decrease in the dielectric constant of the samples.



(a)

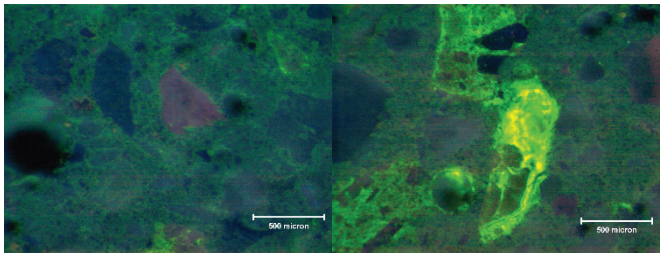


(b)

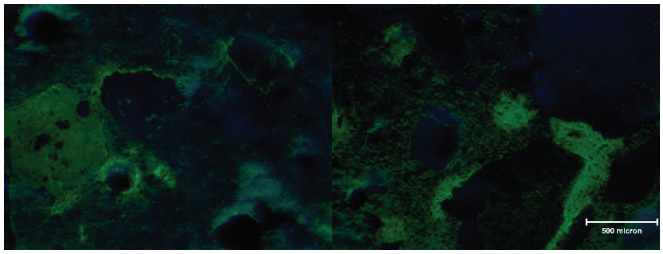
FIGURE 1. Dielectric constant measurements, a) permittivity, b) loss factor.

Comparing the measurement results of the six mortar samples, ultimate (last day) permittivities are different among the samples to some extent. To investigate whether this difference in permittivity values is due to ASR gel or the remaining moisture, corresponding loss factors results were examined. According to Fig. 1b, the (last day) loss factor measurements are almost identical. Since loss factor (compared to permittivity) is more sensitive to the moisture content of the mortars susceptible to ASR [10], the similar values of loss factor (at last day) implies that equivalent amount of moisture (even though very little) is remained within the mortar samples. This is meaningful since the majority of the remaining free water in the samples had sufficient amount of time (~110 days before the last day measurement) to evaporate. Therefore, slight differences in the permittivity measurements may then be attributed to the difference in the materials matrix (or ASR gel formation) rather than the remainder of water in the samples. Consequently, to verify that whether the differences in permittivities are indication of ASR gel, microstructural characterization was performed using UV fluorescence microscopy [13] following the ASTM C856 standard [14].

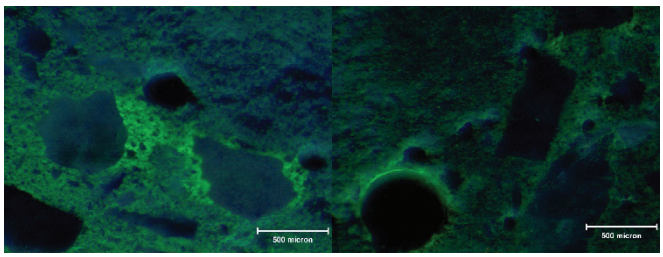
The representative images of six mortar samples (two images per sample) are shown in Fig. 2a-f, where the bright green color in those is the indication of ASR gel.



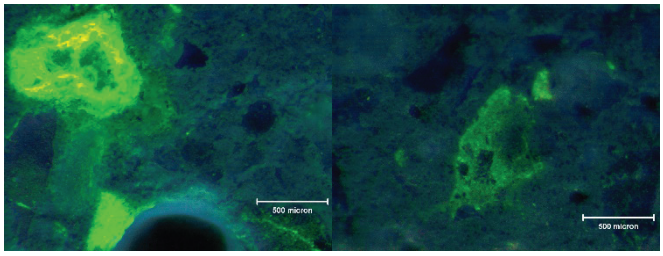
(a)



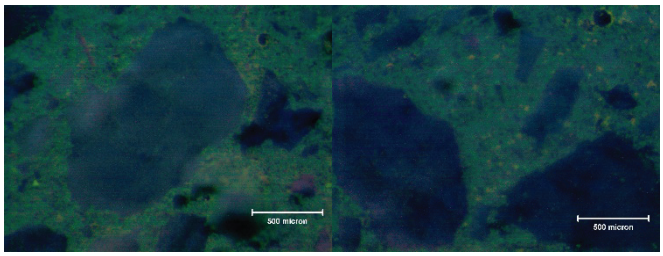
(b)



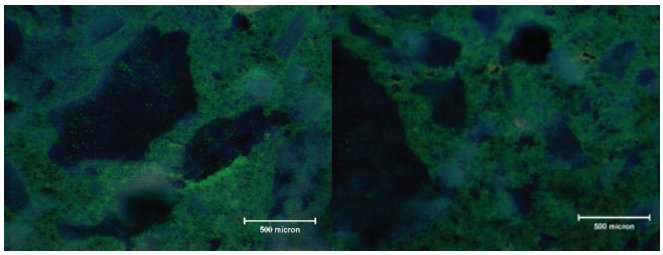
(c)



(d)



(e)



(f)

FIGURE 2. UV fluorescence microscopy images of a) sample 1, b) sample 2, c) sample 3, d) sample 4, d) sample 5, and f) sample 6.

According to the storage conditions of samples in batch #1, since the exposure time to hot and humid conditions is longer for sample 2 than sample 1, more ASR gel formation in the former than the latter is expected. However, the samples have similar permittivities, which suggests the same amount of ASR gel formation. In addition, as shown in Fig. 2a-b, the UV fluorescence microscopy images of samples 1 and 2 show similar level (qualitative) of ASR gel formation. This corroborates that permittivity measurements are correlated to ASR formation.

In batch #2, since sample 4 is further exposed to humid conditions after 180 days storage, more ASR gel formation in that sample than sample 3 is expected. Moreover, according to Fig. 1a for batch #2, the permittivity measurements also show a discrepancy between the permittivity of the two samples, suggesting different material properties. Furthermore, as shown in Fig. 2c-d, the UV fluorescence microscopy images of those samples also show different amounts of ASR gel formation in the samples, where more ASR gel is formed in sample 4 compared to sample 3. Hence, UV fluorescence microscopy corroborates that the difference in permittivities is an indication of ASR gel. In addition, it is unlikely that the difference in the permittivities of sample 4 is due to remainder of moisture for two reasons: first, sample 4 was at ambient condition before the final-day measurement for ~110 days, which should be

sufficient for the sample to lose moisture and reach a similar level of internal moisture as in sample 3. Second, and more importantly, the loss factor measurements of batch #2, in Fig. 1b, show similar values for both sample 3 and 4, indicating the same amount of moisture content in those samples. Hence, the difference in the permittivity of sample 3 and 4 may be attributed to ASR gel formation, rather than variations moisture content independent of that reaction. In batch #3, since samples 5 and 6 were stored at low humidity environment (i.e., ambient conditions), no ASR gel formation is expected. However, Sample 6 was exposed to humid conditions after 180 days to investigate the effect of ASR gel formation on the complex dielectric constant of mortar at later ages. Comparing results of these two samples, permittivity measurements in Fig. 1a show slightly different values for samples 5 and 6. Similarly, the UV fluorescence microscopy images of those sample in Fig. 2e-f, also show small differences in the (apparent) ASR gel. Similar to batch #2, UV fluorescence microscopy images corroborate that the difference in permittivity of the samples appears to be an indication of ASR gel. The same reason (similar measured loss factor) as in batch #1, #2, holds true for this set of samples, supporting that the difference in permittivity measurements are indication of different amount of produced ASR gel rather than different moisture content of the samples. It is important to note that in the absence of quantitative data (i.e., image analysis or expansion) the UV fluorescence images only provide qualitative information.

CONCLUSION AND FUTURE WORK

Three sets of mortar samples were cast with potentially ASR-reactive aggregate source, exposed to different environmental conditions, and their long-term dielectric constants were measured temporally. Through dielectric constant measurements, although mortar samples showed similar loss factor at the end of the experiment, their permittivities were slightly different. Similar values of the measured loss factor suggested that the difference in permittivities could be mainly related to the amount of produced ASR gel, and not variations in internal moisture. To verify this, UV fluorescence microscopy images of samples were obtained. Images qualitatively showed different amount of ASR gel formation, corroborating the hypothesis that the difference in the permittivities are directly related to ASR gel formation. Recently, it was shown that ASR formation can be better tracked through loss factor measurements during the early stages of cement curing process [10], [15]. However, in this investigation, it was shown that the amount of produced ASR gel in long-term (once the overwhelming effect of the internal moisture diminishes) can be better determined through permittivity measurements.

To further validate the findings of this investigation, amount of ASR gel in each sample needs to be quantified. This requires acquiring several images of a single sample, apply quantitative image analysis to assess the amount of ASR gel in each sample. Having accomplished the proposed future work, the capability of microwave materials characterization techniques can be further explored to find out a more precise figure of merit in ASR evaluation. These findings and the previous efforts in ASR characterization, collectively, can be further utilized in future pertinent investigations to develop a robust nondestructive microwave technique in evaluation of ASR formation in cement-based structures.

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