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ABSTRACT

This research presented a comprehensive study on the interaction between steel-reinforced cement paste and microwave energy using a single-mode rectangular waveguide. The mechanisms of changes in the adaptive dielectric properties of reinforced cement pastes to predict how these properties are altered when microwave energy were investigated. The structural characteristics of microwave-cured cement paste will be identified. The obtained results show that dielectric properties are relatively high and remain constant during the dormant period. After this period, the hydration reaction resumes and dielectric properties decrease rapidly. Further with the use of microwave heating, the temperature increased monotonically among the positions of measurement during the microwave-curing process. The typical micrographs of the microwave-cured paste at the age of 4 hours after mixing, 28 days after curing in lime-saturated deionized water, and when subjected to microwave energy showed that the samples consisted of hydrated phases and pores, as well as cores of $Ca(OH)_2$ dendrite crystals, calcium silicate hydrate (C-S-H), and granular structure.

KEYWORDS: Interaction, Microwave energy, Microstructural, Rectangular waveguide, Reinforcing steel, Cement pastes

1. Introduction

Heating by microwave energy has been widely used in research and industrial applications [1,2] such as thawing frozen food [3], combustion synthesis [4], wood [5,6], pyrolysis [7], decontamination of surfaces [8], etc. In order to be suitable for microwave heating the material in question must be a dielectric, and most current applications involve porous media composed of a solid phase containing a second fluid phase within the internal voids. Cement paste may be classed as one of these materials.

Concrete is the predominant construction material with annually ½ cubic meters used per capita worldwide [9]. One disadvantage to concrete is the slow development of strength under normal temperatures and pressures. Various accelerated-curing methods have been invented to enhance the rate of strength development, including application of external heat and pressure in an autoclave. However, the low thermal conductivity (0.8 to 1.28 W/m.K) of cement paste reduces heat transfer during curing. Microstructural development in cement paste is dependent on the uniformity of heat liberation from hydration and from external heating, and a steep temperature gradient from the external heat source to the inner regions of the component results in crack formation within the internal structure. In particular, cement paste structures containing reinforcing steel are susceptible to fracture, substantially reducing the service life of the component. Thermal gradient problems may be reduced by using an internal heating process such as microwave curing.

Microwave heating of steel-reinforced cement paste is a relatively new field that will expand in importance in the near future. In particular, research is necessary to understand curing mechanisms with adaptive dielectric properties and to characterize the heat and mass transfer properties of cement paste materials.

2. Experiments

2.1 Materials used

Type I Portland cements in accordance with ASTM C150 [10] were used throughout this test. Deionized water with a pH 7.5 was mixed in specific proportions by controlling water-cement ratios (w/c) by mass of 0.25, 0.38, and 0.45.

2.2 Testing procedures

In order to measure the dielectric properties of the paste at water-cement ratios (w/c) by weight of 0.25, 0.38, and 0.45, it was necessary to use a vector network analyzer (VNA). To measure the dielectric properties of cementitious materials at a frequency of 2.45 GHz, a network analyzer with an open-ended coaxial probe, as shown in Figure 1, was used. The analyzer consisted of a coaxial cavity; microwave reflectometer; 3.5-mm coaxial cable; 3.5-mm female calibration; and short-, open-, matched-load software. The coaxial cavity characterizes measurement in the range of 1.5 - 2.6 GHz with precision not more than 2% of the dielectric constant and 5% of the dielectric loss factor. The measured sample should be assumed; i.e., it should be assumed to have infinite size, non-magnetic material, isotropic and homogeneous properties. In addition, the coaxial cavity must be in contact with the sample under test (MUT). The Nicholson-Ross-Weir conversion process [11,12] was used to calculated dielectric properties. After the cementitious material had been mixed and placed in the mold, it was wrapped in Styrofoam that was 5.0 mm thick in order to protect it from heat loss. Both dielectric properties and semi-adiabatic temperature using a data logger with thermo-couple (Type K) was simultaneously recorded every 180 and 15 minutes, respectively. However, in order to eliminate the effect of the thermo-couple embedded in microwave radiation, three samples were separately tested for dielectric properties and three

for temperature rise.

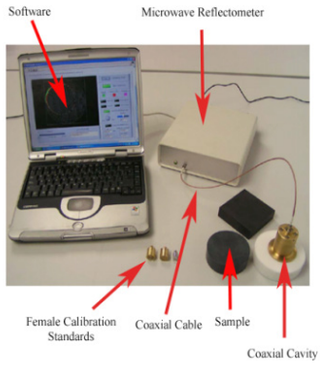

Figure 1 A network analyzer (open ended probe)

2.3 Microwave curing setup

For accelerated curing, the microwave system used was a monochromatic microwave at a frequency of 2.45 GHz, as shown in Figure 2.

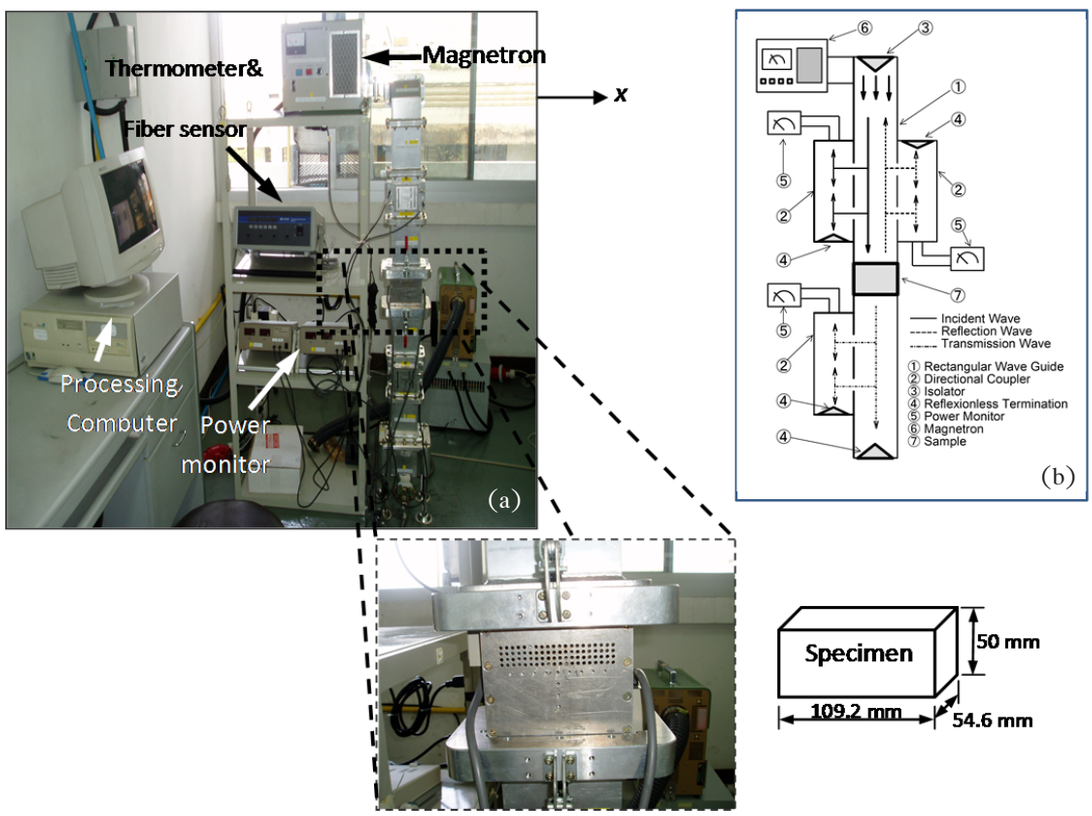

Figure 2 (a) Experimental set up and (b) schematic showing direction of microwave components (Incident wave, reflected wave, and transmission wave)

Microwave energy was generated by a magnetron and transmitted directly along the propagation direction (+z) of a rectangular wave guide toward a water load situated at the end of the waveguide to ensure that a minimal amount of microwave energy would be reflected back to the sample. A warming water load was circulated through the cooling tower in order to reduce the temperature in the water load system.

A cement paste sample was arranged perpendicular to the propagation direction (Figure 3). A Type K thermo-couple with a 0.1 mm diameter was inserted at the center of the sample for the purpose of monitoring the temperature rise. During a 10-min period of microwave heating, the output of the microwave magnetron was controlled at 50, 100 and 150 W. The microwave plane wave traveled directly along the wave guide and made contact with the sample surface; the wave was then reflected and transmitted. By using a wattmeter, incident, reflected and transmitted waves were monitored.

For the specimens subjected to microwave curing, the delay times at 30 minutes after mixing, at the initial setting time, and at the final setting time are determined by the present study for microwave application at the following power levels and durations: power levels of 50, 100, and 150 watt and times of 0, 10, 20, and 30 minutes.

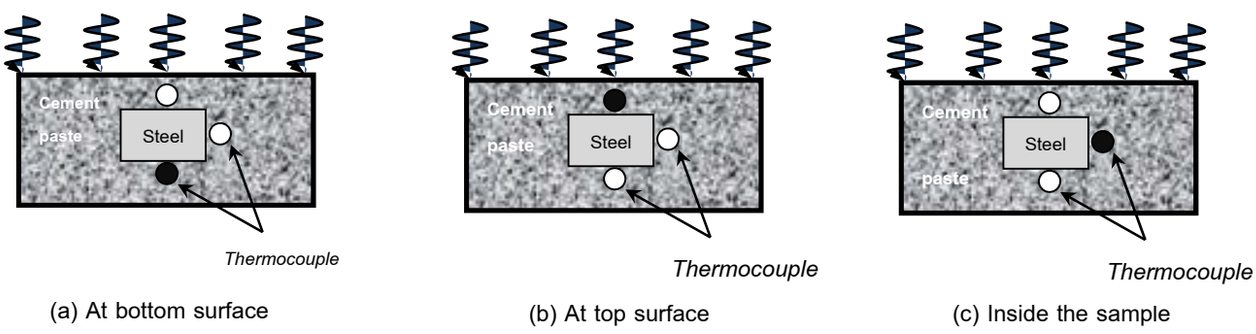

Figure 3 The positions of thermocouples.

2.4 Equipment and procedures

2.4.1 Scanning Electron Microscope

A Scanning Electron Microscope (SEM), specifically an International Scientific Instruments ISI-130 electron microscope, was used to determine the microstructure and morphology of the samples. A dual-stage, dual-screen microscope, it had five lenses and used an energy x-ray (EDX) analysis system. The maximum practical resolution was approximately 100,000 times. The specimens were glued onto a sample stub using carbon tape and then placed in a vacuum chamber and sputtered with gold for approximately 40 minutes at a 75 voltage. The gold film created a route by which the electrons could be conducted off the surface of the sample; otherwise, the accumulation of electrons on the sample surface would have led to charging and a fuzzy picture. The image was displayed on two 30 cm cathode ray tubes.

2.4.2 Powder X-ray Diffraction (XRD)

The crystalline phase identification of the various samples was performed on a Scintag X-ray Diffractometer. This differactometer is equipped with a copper target x-ray source, monochromator, and Tl-drifted NaI scintillation detector. Dried-powder samples were packed into a cavity of a zero-background quartz slide and placed on a goniometry. Most of the subsequent scans were taken from 0 to 70^0 2Θ at a rate of 2^0 2Θ per minute. The diffractometer is controlled by a

VAX 3100 mini-computer and contains a matched software that can be used to display the data on the computer screen and through which data manipulation can be performed. The raw data can be downloaded into a personal computer and the results printed on either a Hewlett Packard plotter or a laser printer. This software package can distinguish between amorphous and crystalline peaks and between $K\alpha_1$ and $K\alpha_{12}$ peaks. In all cases, the raw data used contained some information pertaining to amorphous phases, in the form of humps in the spectra, which would otherwise have been stripped away by the software.

3. Results and discussion

3.1 Dielectric properties

Figure 4 shows the evolution of dielectric properties and the simultaneous temperature rise of cement pastes. It can be observed that the dielectric properties at the initial stage are relatively higher in comparison with the later stage; they also increase with the increasing water content (higher w/s) of the cement pastes. This is due to the fact that immediately after contact has been made between water and cement, they start to react and then Ca^{2+}, OH^-, and SO_4^{2-} ions dissolve into the system [13]. In addition, during the dormant period, the dielectric properties change very little because the chemical composition of the aqueous phase remains nearly constant.

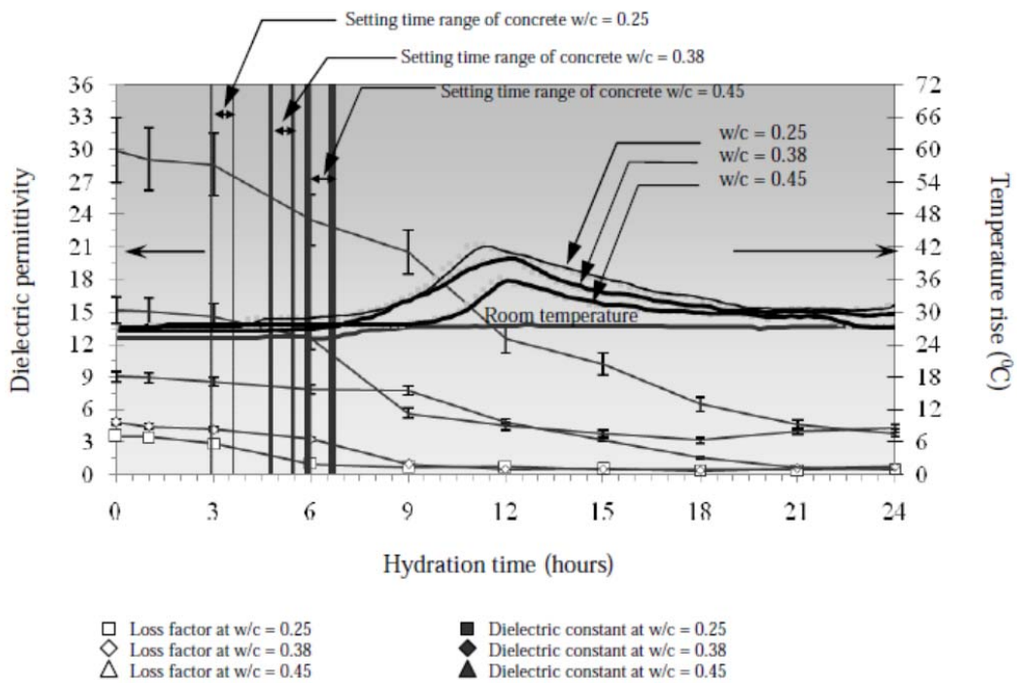

Figure 4 Dielectric permittivity of pastes with different w/c.

Similarly, relative dielectric properties also appear to be affected by the temperature rise. The lower temperature (higher w/c) leads to enhance these properties. This is due to a reduction in water-to-cement-ratio that accelerates hydration and results in higher temperature. Especially, in the accelerated period of the pastes at which it has the highest rise in temperature corresponding to the decreasing relative dielectric properties.

3.2 Microwave heating

3.2.1 Temperature rise

The effect of water-to-solid ratios on microstructures characteristics of the reinforced pastes subjected microwave energy is presented. The pastes used were proportioned at w/c ratios of 0.25, 0.38 and 0.45. After mixing and molding, they were cured at room temperature by wrapping with polyethylene plastic until the delay time (time after mixing until introducing microwave energy with a single-mode cavity) for 30 minutes The temperature profile and microwave power of 100 watt with a specific application time 30 minutes of the pastes is shown in Figure 5. With limitation of the microwave equipment used, it can only adjust manually the steps of microwave power level such as 50, 100, 150,..., 3000 watt, therefore the optimal microwave levels with the aforementioned research should be set at 100 ± 5 watt.

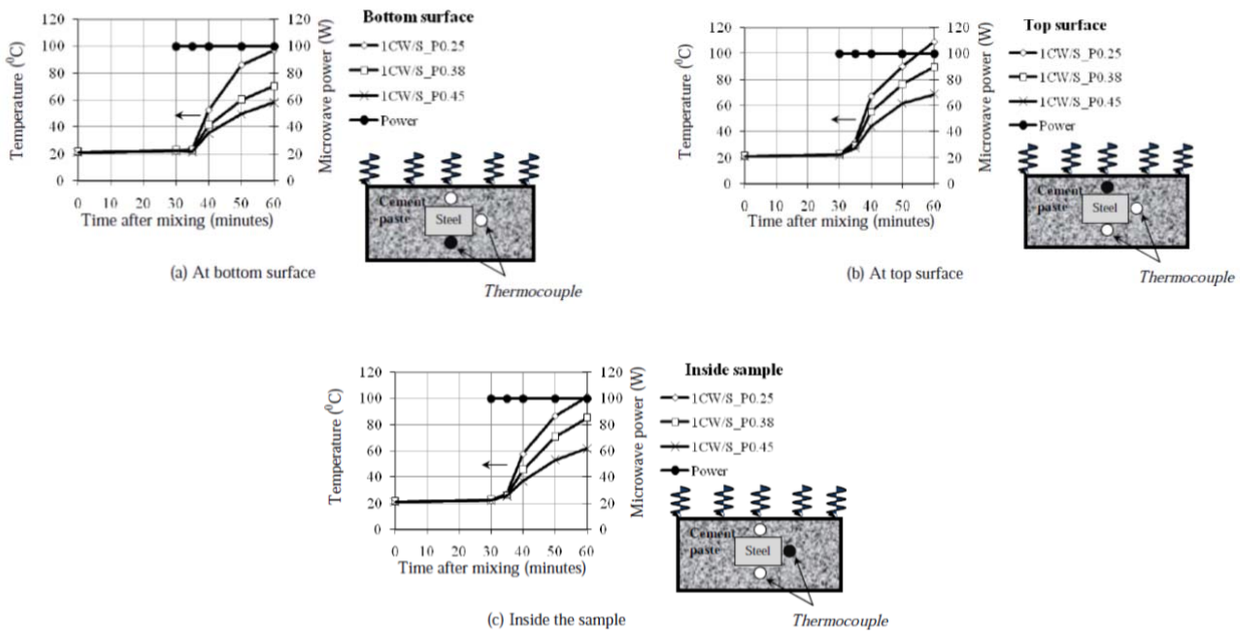

Figure 5 Temperature and power history during applying microwave energy of various cement pastes with different water-cement ratios

Temperature profiles obtained from averaging the five monitored data at bottom surface (Figure 5 (a)), top surface (Figure 5(b)) and at the middle (Figure 5(c)). The temperatures increase monotonically among the positions of measurement during the microwave curing process and reach a maximum of 105^0C at the bottom surface of the cured cement paste. Significantly, the paste at lower water-to-cement ratio experiences high temperature rise, or 0.25 > 0.38 > 0.45. This is because of two inclusive effects; (i) heat liberation from hydration reaction is increased as low content of water in the system, and (ii) heat from interaction between the microwave energy and internal water leading to superposition of them. In other words, the addition heat from microwave energy can change the kinetics of hydration in accordance with Arrhenius's law.

When comparison the obtained temperatures at three positions, the bottom of the cured specimen has highest temperature levels than those of other sides. It may be due to that fact that during temperature rise, the water at the top side of specimen may evaporate, so the temperature was dropping gradually, while at the bottom the evaporation of water is

difficult. As a result the heat accumulates on the specific side providing temperature increase with high rate than the other sides.

3.2.2 Characteristics of microwave-cured pastes

The typical micrographs of the 0.38-w/c paste at the age of 4 hours after mixing, 28 days after curing in lime-saturated deionized water and subjected to microwave energy are shown in Figure 6.

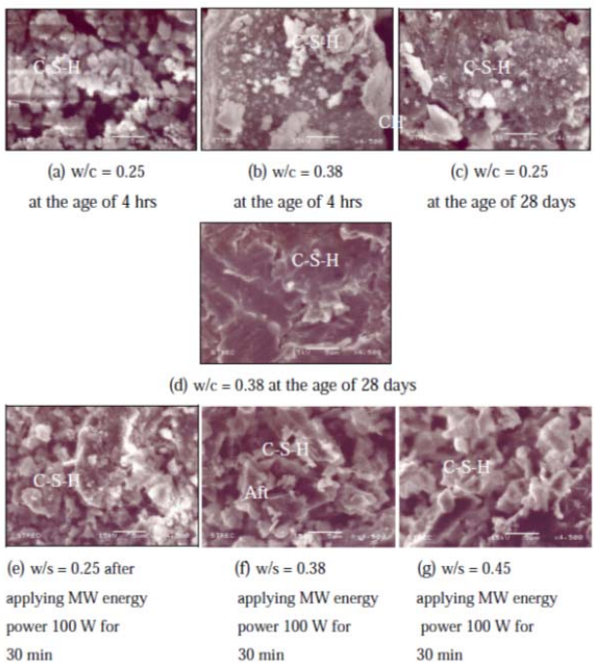

Figure 6 Micrographs of various cement pastes with different water-to-solid ratios at 4 hours subjected to lime - saturated deionized water curing and microwave energy.

It is clearly seen from Figure 6 that the samples consist of hydrated phases and pores, as well as cores of $Ca(OH)_2$ dendrite crystals or other crystals (marked CH), calcium silicate hydrate (C-S-H), and granular structure. Furthermore, some ettringite (Aft) is found in case of specimens were cured by microwave energy. It can be described that in the early stages of reaction of the 27°C sample, very small (about l μm) irregularly-shaped ettringite was formed; but at the same curing time, needle-like ettringite had already formed in 60°C samples.

X-ray diffractometry was used to determine the degree of crystallinity of the hydrated cement products and the existence of crystalline coexisting phases. Figure 7 shows x-ray patterns of the hydrated products in the pastes of 0.38-w/c after applying microwave power 100 watt for 30 minutes. The phases identified include calcium silicate hydrate (Ca_3SiO_5), calcium hydroxide ($Ca(OH)_2$), residual lime (CaO) and Xenotile ($Ca_6(SiO_3)_6(H_2O)$).

Regarding the effect of w/c on the phase characteristics of 0.25, 0.38 and 0.45 after applying microwave power of 100 watt for 30 minutes as shown in Figure 6, it is found that the calcium silicate hydrate (Ca_3SiO_5), calcium hydroxide ($Ca(OH)_2$) phases are similar.

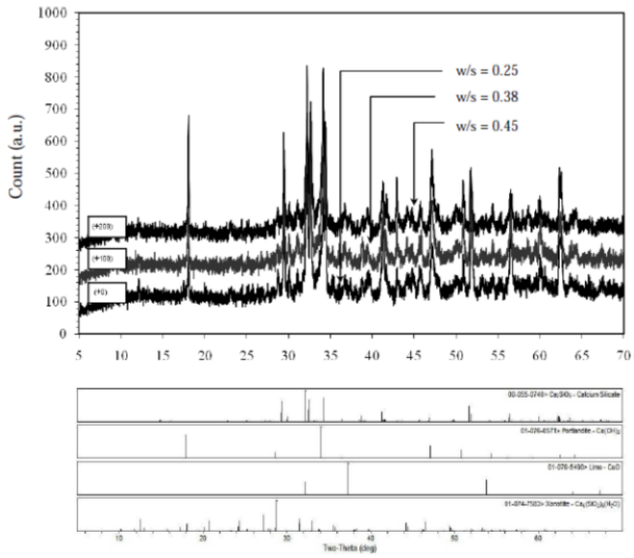

Figure 7 X-ray diffraction of the pastes of 0.25, 0.38 and 0.45 after applying MW power of 100 watt for 30 minutes.

4. Conclusions

The microwave system used to transfer microwave energy in order to heat or cure the reinforced pastes, it can be summarized as follows:

- The dielectric permittivity of reinforced cement-based materials is affected by the initial water-to-cement mass ratio. It should be noted here that the change in the dielectric permittivity of cement-based materials, therefore, is relatively high and remains constant during the dormant period. However, the change in the dielectric permittivity decreases rapidly when the hydration reaction resumes, and it continues to decrease during the acceleratory period.
- The temperature increased monotonically among the positions of measurement during the microwave-curing process. The typical micrographs of the microwave-cured paste at the age of 4 hours after mixing, 28 days after curing in lime-saturated deionized water, and when subjected to microwave energy showed that the samples consisted of hydrated phases and pores, as well as cores of $Ca(OH)_2$ dendrite crystals or other crystals (marked CH), C-S-H, and granular structure.

5. Acknowledgement

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