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Hardened Cement Paste Subjected to Microwave Energy: Heat Transfer Behavior and A Relationship of Strength Development and Maturity

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ABSTRACT

Microwave energy is an energy source for heating (without phase change) and drying (with phase change) dielectric materials. At present, it has been implemented and developed continuously to cure cement-based materials, especially at early age for accelerating the rate of strength development. With the advantages of heating mechanism that generates volumetric inside out, it can be replaced the conventional curing method. This paper presents a point of view of a relationship between heat generated by microwave energy and strength development of cement-based materials (cement plus water plus void). The concept of this relationship is to link microwave energy generating heat inside the processed cement paste and strength development by using the maturity (temperature history) method. The parameters studied include water-to-Portland cement Type I ratio by weight (w/c = 0.40), microwave at the frequency of 2.45 \pm 0.05 GHz, power of 1000 Watts and 15 minutes for time of application. The time before applying the energy is kept constantly at 24 hours after contacting cement particles and mixing water. Furthermore, a twodimensional numerical model is developed to predict power absorption distribution and temperature distribution in cement paste. From the test results, it was found that an increase of microwave power leads to increasing the rate of temperature rise and maximum temperature, and decreasing the rate of moisture contents of cement paste and concrete. Besides, the later age compressive strength of concrete decreased when increasing the power of microwave

Keywords: Hardened Cement paste; Heating; Microwave

1. Introduction

A conceptual development that changes the world is sustainable development. Based on this concept, it is combining the needs of the present without compromising the ability of future generations to meet their own needs. Thus, regarding to this change has resulted in the forms of energy use emphasizing on friendly to environment and without non-valuable by-products such as a clean energy form of microwave.

Microwave is a form of energy responding this purpose. Microwave is a part of electromagnetic band having operating frequencies in a range of 300 MHz-300 GHz as shown in Fig. 1. It are taken into consider as a powerful

heating source. Therefore, microwave processing is usually accomplished at frequencies of 915 \pm 13 MHz or 2,450 \pm 50 MHz (*S-band*) for industrial, scientific and medical applications [1-4].



Fig. 1 Electromagnetic spectrum (Source: http://hyperphysics.phy-astr.gsu.edu/)

A term of microwave heating is equally applicability to microwave systems in both cases; the heating is due to the fact that dielectric materials which are a material with a small but finite electrical conductivity absorb energy when it is placed in a high frequency electric field. Consequently, electrically dipole polarization and conduction will be generated within dielectric materials which are composed of polar molecules with positive \oplus and negative \square poles [5]. These orderly dispersed polar molecules vibrate instantaneously and violently in correspondence to the alternative high frequency electric field. It is necessary to overcome the resistance of molecular attraction and motion, as when friction generated heat, as a result the temperature of the material is evaluated simultaneously [6-7].

Can microwave heating be applied in concrete industry? The answer based on theoretical feasibility, is yes. It was proved by numerous research groups [8-11] both experimentally and numerically. However, some points of curing by microwave are not deeply taken into consideration *this is*, heat and mass transfer mechanisms taking place within concretes. Therefore, this research focuses mainly on characterization of hardened cement paste subjected to microwave energy in a point of view of heat transfer behavior and a relationship of strength development and maturity. This paper is subsequent part for confirming the paper which was submitted to the Int. Comm. in Heat and Mass Transfer Journal.

2. Experimental program

2.1 Sample preparation

The samples studied were made with 24-cement paste specimens with dimensions of 110.0 cm x 54.61 cm x 5.0 cm. By mixing Portland cement Type I (Blaine Fineness of 3,250 cm²/g) together with tap water which kept constantly the water-to-cement ratio (w/c) of 0.40 and 1.0% by volume of void content conforming to the ASTM C 305 standard [12], the paste was poured into moulds. Plastic sheet was used to cover of each mold to protect evaporating of water from the specimens. For $23\frac{1}{2} \pm \frac{1}{2}$ hours after casting, the specimens were demolded, and then half of them were cured with microwave energy and other specimens were cured in water. By curing with microwave using rectangular wave guide for 15 minutes with a constant power of 1000 watts. Whereas water curing, a range of temperature and relative moisture were controlled of 25.0 ± 2.0 degree Celsius and 60.0 ± 5.0 % respectively. The compressive strength of cement paste specimens was tested at the ages of 3, 7, 14, and 28 days conforming to the ASTM C 39 [13].

2.2 Experimental configuration

The microwave system was a TE₁₀ mode monochromatic wave of operating at a frequency of 2.45 ± 0.05 GHz as shown the experimental apparatus in Fig. 2. Microwave energy was generated by a magnetron and transmitted directly along the z-direction of the rectangular wave guide with inside dimensions of 110 mm x 54.61 mm toward a water load that was situated at the end of the wave guide. The water load (lower absorbing boundary) was a part of system for ensuring that only a minimal amount of microwave was reflected back to the sample due to absorb by water. The warm water load was circulated passing through the cooling tower to reduce the temperature in the water load system.

A cement paste specimen is arranged and constrained fitly in perpendicular to direction of irradiation via a rectangular wave guide for eliminating the effects of transitional contact surface of the wall of waveguide and specimen surface. During the experiment, output of magnetron was adjusted at specified power (1000 Watts). The plane wave of microwave travels directly along the waveguide and contacts to the cement paste specimen surface and then was reflected and transmitted. By using a wattmeter, incident, reflected and transmitted waves were measured.

In addition, fiber optic with collective data logger used to detect temperature variation inside of the specimen.



Fig. 2 The microwave experimental apparatus

3. Mathematical modeling and numerical solution

3.1 Assumptions of the cement paste specimen

(a) In fact, at the time of $23\frac{1}{2} \frac{1}{2}$ hours, the hydration reactions of the cement paste progress continuously [14,15]. It means that heat liberation is produced simultaneously as shown in Fig. 3. However, in order to simplify of modeling, heat liberated from hydration reactions is neglected for eliminating coupling phenomena of heat generation by microwave energy.





(b) From the preliminary study that the final setting time of the 0.4-w/c paste is 228 minutes, therefore, it may be confirmed that the structure of calcium silicate hydrates which were a main structure for gaining the setting and hardening the strength in cement paste was formed completely.

3.2 Analysis of electromagnetic distribution inside a rectangular waveguide

A plane wave of electromagnetic in TE_{10} mode is taken into account for calculating electric-magnetic fields. Since microwave of TE_{10} mode propagates uniformly in *y*-direction, the electromagnetic field can be considered in two-dimensional model on *x*-*z* plane (Fig. 4). Thus, such correspondent electromagnetic and temperature fields can be contemplated in two-dimensional model. Fig. 5 illustrates the physical model for the microwave heating of the cement paste using rectangular wave guide. The model proposed has assumptions as follows: [17-19]

(a) The composition materials of cement paste materials (cement plus water) are non-magnetic materials.

(b) The electrical properties of the walls of rectangular wave guide are perfect conductors that can reflect the wave completely.

(c) The absorption of microwave energy by air in rectangular wave guide is negligible.

(d) The effect of the specimen container that made of polyethylene on the electromagnetic and temperature fields can be neglected.



 Fig. 4 The electromagnetic field
 Fig. 5 Physical model

 distribution on x-z plane
 Fig. 5 Physical model

In order to analyze the behavior of electric and magnetic fields in the wave guide, a set of fundamental equations governing the fields Maxwell's equations are used. By using the assumptions, the electromagnetic field can be written in term of the component notations of electric and magnetic field intensities [19].

$$\frac{\partial \bar{E}_{y}}{\partial z} = \mu \frac{\partial \bar{H}_{x}}{\partial t}$$
(1)

$$\frac{\partial \bar{E}_{y}}{\partial x} = -\mu \frac{\partial \bar{H}_{z}}{\partial t}$$
⁽²⁾

$$-\left(\frac{\partial \bar{H}_z}{\partial x} - \frac{\partial \bar{H}_x}{\partial z}\right) = \sigma \bar{E}_y + \varepsilon \frac{\partial \bar{E}_y}{\partial t}$$
(3)

where, \vec{E} and \vec{H} denote electric field intensity in (volt per meter (V/m)) and magnetic field intensity (ampere per meter (A/m)), respectively. The subscripts (*x*, *y* and *z*) represent components of vectors in *x*, *y* and *z* direction, respectively. Further, permittivity or dielectric constant, \mathcal{E} (farads per meter, F/m, \mathcal{E}_{o} is permittivity of free space (8.854 x 10⁻¹² F/m) [20], magnetic permeability, μ and electric conductivity, σ are given by

$$\varepsilon = \varepsilon_0 \varepsilon_r, \ \mu = \mu_0, \ \sigma = 2\pi f \varepsilon \ (tan\delta) \tag{4}$$

Due to the compositions of cement paste are non-magnetic properties. The materials effect is negligible, which is true for most dielectric materials used in microwave heating, the magnetic permeability (μ) can approximate by μ_0 in the free space ($4\pi \times 10^{-7}$ H/m).

The boundary conditions associated with the physical model as shown in Fig. 5, are as follows [19]:

(a) Perfectly conducting boundaries; boundary conditions on the inner wall surface of a rectangular waveguide are given by using Faraday's law and Gauss theorem:

$$\vec{E}_t = 0, \ \vec{H}_n = 0 \tag{5}$$

(b) Continuity boundary condition; boundary conditions along the interface between different materials, for example between air and dielectric material surfaces, are given by using Ampere's law and Gauss theorem:

$$\vec{E}_{t} = \vec{E}_{t}^{'}, \vec{H}_{t} = \vec{H}_{t}^{'}, \vec{D}_{n} = \vec{D}_{n}^{'}, \vec{B}_{n} = \vec{B}_{n}^{'}$$
(6)

(c) Absorbing boundary condition; at both ends of the rectangular wave guide, the first order absorbing conditions is applied:

$$\frac{\partial \vec{E}_{y}}{\partial t} = \pm \upsilon \frac{\partial \vec{E}_{y}}{\partial z}$$
(7)

The symbol \pm represents forward or backward waves and $\,\mathcal U\,$ is phase velocity of the microwave.

(d) Oscillation of the electric and magnetic flied intensities by magnetron; incident wave due to magnetron is given by the following equations:

$$E_{y} = E_{yin} sin\left(\frac{\pi x}{L_{x}}\right) sin(2\pi ft)$$

$$E_{yin} = \left(\frac{\pi x}{L_{x}}\right)$$
(8)

$$H_{x} = \frac{E_{yin}}{Z_{H}} \sin\left(\frac{\pi x}{L_{x}}\right) \sin\left(2\pi ft\right)$$
(9)

 Z_{H} is the wave impedance defined as:

$$Z_{H} = \frac{\lambda_{g} Z_{I}}{\lambda_{0}} = \frac{\lambda_{g}}{\lambda_{0}} \sqrt{\frac{\mu_{0}}{\varepsilon_{0}}}$$
(10)

The finite difference time domain method is applied to predict the electric and magnetic fields, the leapfrog scheme are implemented to set of Maxwell's equations. The electric field vector components are offset one half cell in the direction of their corresponding components, while the magnetic field vector components are offset one half cell in each direction orthogonal to their corresponding components. The electric and magnetic field are evaluated at alternative half time steps. For TE₁₀ mode, the electric and magnetic field components are expressed the total field FDTD equations as:

$$E_{y}^{n}(i,k) = \frac{1 - \frac{\sigma(i,k)\Delta t}{2\varepsilon(i,k)}}{1 + \frac{\sigma(i,k)\Delta t}{2\varepsilon(i,k)}} E_{y}^{n-1}(i,k) + \frac{1}{1 + \frac{\sigma(i,k)\Delta t}{2\varepsilon(i,k)}} \frac{\Delta t}{\varepsilon(i,k)} \left\{ \frac{-\left(H_{z}^{n-1/2}(i+1/2,k) - H_{z}^{n-1/2}(i-1/2,k)\right)}{\Delta x} \right\}$$

$$\frac{\left(H_x^{n-1/2}(i,k+1/2) - H_x^{n-1/2}(i,k-1/2)\right)}{\Delta z}$$
(11)

$$H_{x}^{n+1/2}(i,k+1/2) = H_{x}^{n-1/2}(i,k+1/2) + \frac{\int \Delta t}{\mu(i,k+1/2)} \left\{ \frac{E_{y}^{n}(i,k+1) - E_{y}^{n}(i,k)}{\Delta z} \right\}$$
(12)

$$H_{z}^{n+1/2}(i+1/2,k) = H_{z}^{n-1/2}(i+1/2,k) - \frac{\Delta t}{\mu(i+1/2,k)} \left\{ \frac{E_{y}^{n}(i+1,k) - E_{y}^{n}(i,k)}{\Delta x} \right\}$$
(13)

3.3 Heat transfer analysis

The temperature of processed cement paste under soaking with microwave can be obtained by solving the heat conduction transport equation with the microwave power included as a local electromagnetic heat generation term:

$$\frac{\partial T}{\partial t} = a \left(\frac{\partial^2 T}{\partial x^2} + \frac{\partial^2 T}{\partial z^2} \right) + \frac{Q}{\rho \cdot C_p}$$
(14)

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where, τ is temperature, a is thermal diffusivity, ρ is density and c_p is heat capacity at constant pressure. The local electromagnetic heat generation term Q depends on the electric field distribution defined as:

$$Q = 2\pi f \varepsilon_0 \varepsilon_r (tan\delta) E_y^2 \tag{15}$$

The initial condition of multi-layered materials defined as: $T = T_0$ at t = 0. The boundary conditions for solving heat transport equation [19].

The heat transport equation (Eq. 14) is solved by the method of finite differences. The spatial and the temporal terms are approximated using finite difference equations for electromagnetic field and temperature field. Ratanadecho, P. [9] discretized heat transport equation are solved on this grid system. The choice of spatial and temporal resolution is motivated by reasons of stability and accuracy. To insure stability of the time-stepping algorithm Δt must be chosen to satisfy the courant stability condition:

$$\Delta t \le \frac{\sqrt{\left(\Delta x\right)^2 + \left(\Delta z\right)^2}}{\upsilon} \tag{16}$$

and the spatial resolution of each cell is defined as:

$$\Delta x, \Delta z \le \frac{\lambda_g}{10\sqrt{\varepsilon_r}} \tag{17}$$

The calculation conditions which correspond to Eqs. (16) and (17), are described as follows: [9]

(a) To ensure that each wavelength of the microwave in the computational domain for a frequency of 2.45 GHz has more than 10 subdivisions in the numerical calculation. Thus, the computational domain is conservatively set such that the spatial resolution of each cell is $\Delta x = \Delta z \leq \lambda_{mg} / 10 \sqrt{\varepsilon_r} \approx 1.0 \text{ mm}$. Thus, the total 110 x 250 cells in computational domain were used in the numerical calculation,

(b) Because of the propagating velocity of microwave is very fast compared with the rate of heat transfer, the different time steps of dt = 1 [ps] and 1[s] are used for the computation of the electromagnetic field and temperature field, respectively. The spatial step size is dx = dz = 1 [mm],

- (c) Number of grid: N = 110 (width) x 250 (length),
- (d) Relative errors in the iteration procedure of 10^{-8} were chosen.

3.4 Analysis of maturity of the heated cement paste

The maturity method uses to determine the strength development of concrete for different evaluated temperature curing method lied on the combined effects of time and temperature. A famous Nurse-Saul maturity (temperature-time factor) function [21]:

$$M(t) = \sum_{0}^{t} (T - T_0) \Delta t \tag{18}$$

where, *M* is maturity index or temperature-time factor [10], [°]c-hours. *T* is average concrete temperature, [°]c. T_o is datum temperature (usually taken to be -10 [°]c). *t* is elapsed time and Δt is time interval in hours.

In addition, the maturity index can be approved based on the Arrhenius equation that used to describe the effect of temperature on the rate of a

chemical reaction. Thus, the equivalent age (t_e) of cement-based materials is set as follows [22]:

$$t_e = \sum_{t=0}^{t=t} e^{\frac{-E}{R} \left(\frac{1}{T(K)} - \frac{1}{T_r(K)} \right)} \Delta t$$
(19)

where, t_e is equivalent age, *E* is apparent activation energy (J/mol), *R* is universal gas constant (8.314 J/mol.K), *T* is the average absolute temperature of concrete during time interval Δt (°K), and T_0 is the absolute reference temperature (23 °K).

One of a simple strength-maturity relationship [23] used the logarithm equation for strength gain under isothermal curing up to equivalent age at 23 $^{\circ}$ C of 28 days.

$$S = a + b \cdot log(M) = a + 2.302 \cdot b \cdot ln(M) \tag{20}$$

where, S is strength at age t, a is strength for maturity index (M = 1), b is slope of the line and M is maturity index.

4. Results and discussion

4.1 Electric field dissipation

Fig. 6 shows the electric field distribution within a cement paste specimen during microwave heating. Due to high lossy material of the paste at the age of 1 day after cement particles and water are mixed together; therefore, a penetrating irradiation of microwave is smaller depth in comparison to the depth of specimen. Consequently, larger part of microwaves is absorbed by the specimen. It is observed from the figure that the resonance of standing wave configuration inside the small specimen is weak.



Fig. 6 A typical electric field distribution rectangular wave guide is filled with cement paste sample

(*t* = 60 sec., Power = 1000 W).

4.2 Temperature distributions

Fig. 7 exhibits temperature in cement paste between heating/curing by microwave energy. The temperature distributions correspond to the electric field distribution in the processed specimen. This is because attenuation of the electric field when travels through the specimen owing to energy absorption and thereafter the absorbed energy are converted to heat resulted in increase the temperature of the heated cement paste. For example, in addition the temperature distributions are shown for time of application of microwave for 15 minutes bring about the maximum temperature approaches to around 224 °C. It is observed that the temperature distributions within the paste display a weak wavy behavior due to the penetration depth of microwave drops dramatically and the wavelength is short. Since the reflected wave from the lower surface of the paste is almost negligible, a weak resonance is formed within the paste.

Regarding to tendency of temperature rise within the cement paste, Fig. 8 shows that temperature rise is increased continuously when the heating time of microwave energy increased. The feature trend of temperature increment consists of high rate at the early age and continuous decrease. It is caused by the effect of penetration depth of absorbed microwave energy and the amount of water content at the surface of the heated specimen. This means that before setting of cement paste, the cement particles settled as gravitational force lead to high porosity or capillary pores with soaked surface near contacting surface to microwave energy.







Fig. 9 shows the strength development of cement paste after

curing by means of microwave energy comparing with water curing. It can be seen that the strengths of cement paste cured by microwave are higher than those for cement paste cured by water. The compressive strengths of both microwave-cured and water-cured cement paste increase quickly at early stage until 7 days. From the figure, the early strength of microwave cured cement paste is clearly superior. The results indicate that microwave application can significantly increase the degree of hydration during a few days, after which the microwave cured specimens and control specimen attain similar degree of hydration. After 7 days, the strength appears to reach to be constant while at the long term the strengths of microwave cured specimens are slightly lower than those for normal cured specimens. This is because microwave cured at short time exhibits less microcracks corresponding to a better micro structure arrangement and the compressive strength is improved [24]. However, after the age of 14 days illustrates crossover behavior of the strength between watercured and microwave-cured cement paste specimen due to high early hydration of cement under over-heating can produce a large amount of very fine calcium silicate hydrate (C-S-H) gel coating the unhydrates, causing hindrance of diffusion and crystallization of the products of hydration reactions and consequent to development of long term-strength [25].



Fig. 9 Comparison of compressive strength development for different curing methods

4.3 Strength-Maturity relationship

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According to Eq. (18)-(20) and the ASTM 1074 [12], it can be rewritten the relationship of strength-maturity index in Eq. (21). Furthermore, Table 1 reports the value of the referred parameters in order to calculate the Strength-Maturity relationship.

$$t_e = \sum_{t=0}^{t=t} e^{\frac{-E(J/mol)}{8.314(J/mol.K)} \left(\frac{1}{(T(^0C)+273)(K)} - \frac{1}{T_r(^0C)+273(K)}\right)} \Delta t (days)$$
(21)

Table 1 The value for calculating the relationship

Initial conditions	Value	Reference
w/c (by weight)	0.4	-
Temperature	30 [°] C	-
Properties of cement	Value	Reference
paste		
absolute reference	23 [°] C	[26]
temperature	23 + 273 [°] K	
apparent activation	E (KJ/mol) = 0.685 x	[27]
energy	(T [°] C) + 18.117	

A relationship are plotted which made of the average compressive strength as a function of the average maturity index (factor). Fig. 10 shows the strength-maturity relationship of the cement paste having water-to-cement ratio equal to 0.40 (by weight) subjected to microwave energy associated with rectangular waveguide. It can be illustrated that a straight line with the coefficient (a = 284.37 and b = 88.188) is the best-fit smooth curve from regression analysis.



5. Conclusion

5.1 Curing by microwave energy associated with rectangular wave guide can be affected the long-term strength development (28 days) of cement paste having w/c = 0.4. This is because microwave cured at long time exhibits more microcracks taking place and high early hydration of cement under over-heating can produce a large amount of fine C-S-H.

5.2 A straight line relationship to describe the strength-maturity of the 0.40-w/c cement paste under microwave energy (by weight) is :

$$Strength^{Test \, days} = 284.37 + 88.188 \ln(Maturity \, Index)^{Test \, days}$$
(22)

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Nomenclature

В	magnetic flux density (Wb/m ²)
D	electric flux density (C/m ²)
E	electric field intensity [V/m]
f	frequency of incident wave [Hz]
Н	magnetic field intensity [A/m]
Q	local electromagnetic heat generation [W/m 3]
Т	temperature [C]
t	time [s]
$\tan\delta$	loss tangent [-]
Z_H	wave impedance [Ω]
Z_{I}	intrinsic impedance [Ω]

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