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Hydration and microstructure of cement-based materials under microwave curing

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HIGHLIGHTS

• Microwave curing improves the early strength when compared to steam curing.

- Microwave curing reduces the pores in the range of >100 nm.
- Microwave curing forms short-rod AFt and diminishes the particle size of CH.
- Microwave curing increases the absorption of S, Mg and K by C-S-H gel.

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ABSTRACT

By reducing the curing time, microwave curing can enhance the productivity, save the capital and decrease the plant areas for precast concrete when compared with the steam curing. Based on the results of 6-h and 24-h compressive strength tests the optimum curing regime was selected. The sample microwave cured using the selected curing regime was then compared against samples cured using (a) normal curing (b) steam curing at 40 °C for 10 h and (c) steam curing at 80 °C for 4 h by performing the compressive strength, XRD, TG-DSC, SEM-EDS and MIP. The results indicate that, compared with the steam curing at 80 °C, microwave curing improves the compressive strength of mortar before the age of 28 days, increases the porosity of mortar slightly, while reduces the pores in the range of >100 nm greatly, forms short-rod AFt and smaller particle size of calcium hydroxide, and increases the adsorption of K, S and Mg by C-S-H gel.

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1. Introduction

There are many advantages when the curing period is reduced, such as productivity improvement, capital saving, and the reduction of workshop area, et al. Thermal curing technique has been used in the precast concrete for a long time. However, as a poor heat conductor, it takes >10 h to complete one curing cycle before demoulding [1]. In addition, concrete strength after 28 days is lower than that under standard curing because the fast early hydration of cement under such a high temperature, which results in the formation of large amounts of very fine C-S-H gel surrounding the unhydrates, and hence causing hindrance of diffusion and further development of strength [2].

High-frequency electromagnetic heating, such as microwave enhanced heating, is able to reduce such heterogeneity due to its

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http://dx.doi.org/10.1016/j.conbuildmat.2016.03.202 0950-0618/© 2016 Elsevier Ltd. All rights reserved. superior penetration depth. Microwave energy is attenuated by the vibration of polar molecules and dipoles and the resulting friction between the molecules generates heats rapidly throughout the concrete. Research by Watson [3] showed that 28-day compressive strength of microwave cured specimens displayed only half the strength compared to normally cured specimens. However, by optimizing the microwave curing parameters, strength can be improved effectively [4–6], making microwave curing a potential alternative method for accelerating cements hydration. Research by Xuequan et al. [2] found an increase in early strength under microwave curing, without any detrimental effect at later ages. Results of the permeability revealed that the specimens under microwave curing were denser than the reference specimens, inferring a reduction in porosity and the action of plastic shrinkage. Studied by Leung et al. showed that type III Portland cement concrete with microwave curing can develop early-age strength (at 4.5 h) and later-age strength (7 day) that compare very favorably with commercially available rapid hardening concrete as







well as concrete containing accelerating admixtures [7]. Makul et al. [8] recently reported that the use of a Cober Electronics industrial microwave generator (2.45 GHz, 6.0 kW) into a multimode applicator, resulted in specimens that exhibited increased strength over those cured under autoclaved and lime saturated conditions, particularly at early ages. In general, the performances of the concrete are influenced by the hydration and microstructure. Microstructural and compositional analysis did reveal differences in the cement specimens based on the applied curing regime [8]. Ettringite (AFt) was found in the microwaved and autoclaved specimens, but not in specimens cured under lime saturated water, where plate-like calcium hydroxide or possibly monosulfate (AFm) was identified by authors. Microwave curing promoted disorder within the C-S-H phase, which provides cement with its long-term mechanical strength. Microwave curing made the C-S-H crystals smaller, fractured and more randomly orientated. And the Xenotile $(Ca_6(SiO_3)_6(H_2O))$ was poorly resolved, suggesting an uncertain and complicated shape, despite being crystalline.

Intrinsic properties of materials affect the way they interact with the electric and magnetic fields of the microwaves. In order to discuss the heating kinetic, in general, non-magnetic material was assumed [8,9]. Therefore, the complex (electric) permittivity comprised with real and imaginary parts can be defined as the relationship expressed in Eq. (1). Where, ε'_r and ε''_r are the real and imaginary parts, and $\vec{i} = \sqrt{-1}$.

$$\mathcal{E}_r^* = \mathcal{E}_r' - \overrightarrow{j} \mathcal{E}_r'' \tag{1}$$

However, the influences of microwave curing on the hydration and microstructure of cement-based materials cannot solely be attributed to excessive material shrinkage and acceleration of hydration resulting from rapid dewatering due to high temperature rises under microwave treatment. In addition, in order to further understand the relationships between the microstructure and the superior performances of cement-based materials cured under microwave, the optimum microwave curing regime was selected. The compressive strength of mortar under the optimum microwave curing regime was compared with that of mortar under normal curing, 40 °C steam curing and 80 °C steam curing. The

 Table 1

 The chemical composition of P.I 42.5 cement (mass%).

hydration was tested by X-ray diffraction analysis (XRD) and thermo gravimetric-differential scanning calorimetry (TG-DSC), the morphology of mortars was measured by scanning electron microscopy-energy dispersive X-ray Spectrum (SEM-EDS). The pore structure of mortars was tested by mercury intrusion porosimetry (MIP).

2. Material and methods

2.1. Material

The chemical composition of P.I 42.5 cement is listed in Table 1. The ISO standard sand conforming to Chinese National Standard GB/T 17671-2005 is used. Specimens with a diameter of 4 cm and the height of 8 cm were prepared by cylindrical molds processed by Nylon with a wall thickness of 4 mm. The water to cement ratio of mortar is 0.45. The sand to cement ratio is 3.00. The water to cement ratio of paste is 0.25.

2.2. Curing regimes

Four curing regimes i.e. normal curing (20 °C, RH 95%), steam curing at 40 °C for 10 h, steam curing at 80 °C for 4 h, and microwave curing were studied. 2-h delay period prior to steam curing was needed. The rates of temperature increase and drop of steam curing were 15 °C/h. The microwave radiation was fixed in the microwave oven with a chamber of 23 L. Eight specimens with molds on a turntable was heated together to radiate homogeneously. The nomenclature of specimens with specific microwave curing methods was listed in Table 2. After the curing in Table 2, all of the specimens were cured in water at room temperature after demoulding.

2.3. Test methods

According to the curing regime of M5, the upper-surface temperature of the specimens in mold were measured before the microwave turning on or after the microwave turning off immediately (in 10 s) using an infrared thermal scanner with its emissivity set to 0.95, which is the optimum to detect concrete temperatures [10]. The temperature history of the mortar under microwave curing is shown in Fig. 1.

The compressive strength was tested by a hydraulic compression testing machine with a loading rate of 2.4 kN/s. For later use in SEM, EDS and MIP, the cores of broken mortar at 1 day and 28 days were collected and put into the ampere botles filled up with absolute ethyl alcohol to terminate hydration. The pastes were vacuum-oven-dried at 40 °C until constant mass and then ground to powder for XRD and TG-DSC tests.

XRD analysis was carried out by using a Rigaku D/max 2550 X-ray diffractometer at a continuous scanning rate of 10° /min with Graphite-monochromatized Cu K α radiation generated at 40 kV and 200 mA. The TG-DSC experiments were mea-

SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	Na ₂ O	f-CaO	Loss on ignition
20.81	4.92	3.41	62.65	2.38	2.65	0.67	0.81	2.01

 Table 2

 The sample numbers and microwave curing regimes.

Number	Curing regimes
Ν	Temperature 20 ± 2 °C; Relative humidity >95%
S40	Steam curing at 40 °C
S80	Steam curing at 80 °C
M1	D60 + M5 + I30 + M5 + I30 + M5
M2	D60 + ML5 + I30 + ML5 + I30 + ML5 + I30 + ML30
M3	D60 + ML5 + I30 + ML30
M4	D60 + ML5 + I30 + ML10
M5	D60 + ML5 + I30 + ML20
M6	D60 + ML5 + I30 + ML15
M7	D60 + L10 + I30 + L10 + I30 + L10 + I30 + L10 + I30 + L10 + I45 + ML15
M8	D120 + ML5 + I30 + ML20
M9	D30 + ML5 + I30 + ML20
M10	D30 + ML5 + I30 + ML20 + I40 + ML15

Note: D represents delay time from the moment adding water into the cement to the first microwave radiation; I represents interval time between two times microwave radiation; M, ML and L represent output power of 440w, 260w and 140w, respectively. For example, D60 + M5 + I30 + M20 means that, at the condition of output power of 440w, 60 min after adding water is needed before the first microwave radiation for 5 min, then shut down the radiation for 30 min, then radiates for 20 min at last.

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