Microstructures and Mechanical Properties of Concretes at Early Age when Subjected to Microwave-accelerated Curing with A Multi-mode Cavity

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Abstract

This paper presents the microstructures and mechanical properties of concretes subjected to microwave energy with a multi-mode cavity by varying microwave power levels. The microstructures of hydration products of 0.38-w/c concretes were examined by scanning electron microscope (SEM) associated with energy dispersive X-ray analysis (EDX) and X-ray diffraction (XRD). Furthermore, compressive strengths of the concretes were also tested. The obtained results shown that the temperature increases monotonically during the microwave curing process. The phases which includes calcium silicate hydrate (Ca_3SiO_5), calcium hydroxide ($Ca(OH)_2$) and xenotile ($Ca_6(SiO_3)_6(H_2O)$) are identified. When cured at elevated temperatures, concretes can develop strength quite rapidly. At the age of 8 hours after microwave curing at a power 180 watt, concrete attained a strength of 6.90 MPa (127.8 % higher than the lime-saturated water-cured concrete); at 24 hours curing the strength is 16.8 MPa, and 7 and 28 days the strengths are 118.9 and 118.5 MPa, respectively.

Keywords: Concretes; Microwave; Multi-mode cavity; Microstructure; Mechanical

1. Introduction

The use of microwave energy to improve the properties of cement-based materials is a relatively new area of research [1-3]. However, this is a growing area of interest because microwave heating has many advantages, including high speed for heat generation, high-energy penetration, instantaneous and precise electronic control, and clean process. Although hydraulic Portland cement-based material offers many usefulness in construction work and is also one of most widely used materials of mankind, it consumes more time to develop in order to satisfying standard strength and elasticity. Thus many techniques and methods have been invented and developed continuously

such as using of high-early strength Portland cement or Type III [4-5], adding an accelerator agent, thermally accelerating method i.e. both high temperature at atmospheric pressure and high pressure (autoclave curing). Unfortunately, thermally cured methods lead to drawback effects to its properties both early age and long term. For example, high temperature at atmospheric pressure encounter lowered long-term strength and associated with serious durability problem which recent knowledge not obviously and wholly understood. The reason might be from an increasing micro-cracking and also occurring of delayed ettringite formation (DEF) as mentioned in a research of Verbeck and Helmuth [6] that rapid acceleration in hydration reaction led to encapsulation of the anhydrous cement grains by a product layer of low porosity, which retarded further hydration. Furthermore, it is well known that hydration products are an insulating (dielectric) material that can transfer heat in low rate and non-uniform consequent to its poor properties. Therefore, if we move heat source from the outside surface of the heated cement-based materials to internal structure by using the interaction between microwave (electromagnetic) and the heated cement-based materials which can be violently interacted with cement-water system resulting in volumetric heat generation [7-9]. This concept will be one of the accelerated curing methods. However, due to Portland cement-water inherently reacts to a complex multi-component system and repletion coupled temperature and composition sensitive hydration product, therefore an investigation of microstructure characterization of Portland cement-based materials subjected to high temperature curing using microwave radiation energy is necessary in order to develop this method.

2. Experimental program

Types I hydraulic Portland cement was used throughout this test. Their chemical compositions and physical properties conformed to the ASTM C150 [5]. Tap water with a pH 7.0 and river sand and crushed limestone rock with fineness moduli of 2.58 and 7.23, respectively, and gradation conforming to the ASTM C33 [10], were mixed in specific proportions as shown in Table 1. The chemical admixture used superplasticizer that conforms to the ASTM C494 [11]; that is, the superplasticizer had a recommended dosage rate of 1000 ml per 100 of a kilogram of cement materials.

The concretes used were proportioned at a w/c ratio of 0.38. The specimens were removed from the mold at 23½ ± ½ hours after the start of mixing and then cured by conventional curing, rapid curing with microwave energy, and rapid curing: autoclave curing. The specimens subjected to conventional curing were immersed in a $Ca(OH)_2$-saturated solution. For microwave curing, 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 multi-mode cavity) for 30 minutes. The appropriate amounts of starting materials were weighed out to the nearest hundredth of a gram on a Mettler PI 1200 balance. A Hobart mixer was used to mix the solids and liquids according to ASTM C305 [12]. Samples were cast as

ϕ 70.0 mm × 40.0 mm cylindrical specimens. The samples were cured by using saturated lime water at 25 ^{0}C, microwave energy with a multimode cavity.

Table 1 Mixtures used (by weight in grams).

Symbol	W/C	Cement	Water	Superplasticizer Type F	Sand	Rock
		(g)	(g)	(g)	(g)	(g)
3CAM0.38_ 1:2.75	0.38	500	190	10	1375	0
3CAC0.38_ 1:1:1	0.38	500	190	10	500	500

This study set up a microwave sintering system, as shown in Fig. 1, that included an industrial microwave generator model S56F manufactured by Cober Electrics, Inc., Stanford Conn., USA. This model can generate microwave energy at 2.45 ± 0.05 GHz and a maximum power of 6.0 kW with a multimode system. This results in a temperature of 2000 ^{o}C being induced in the material to be heated. Moreover, hydrogen, nitrogen, and argon can be controlled using this system. However, the maximum cubical size of the material to be processed is limited to 102 mm.

The microwave apparatus does not provide a real-time monitoring of temperature changes during microwave curing; therefore, the temperature of the sample should be measured at the start and end of the curing process. In order to measure the temperature of the sample subjected to microwave energy, the positions of measurement were determined. The temperature of the top surface and the bottom surface was measured 5 times for each; likewise, the sample was immediately fractured such that temperature within it was also measured 5 times.

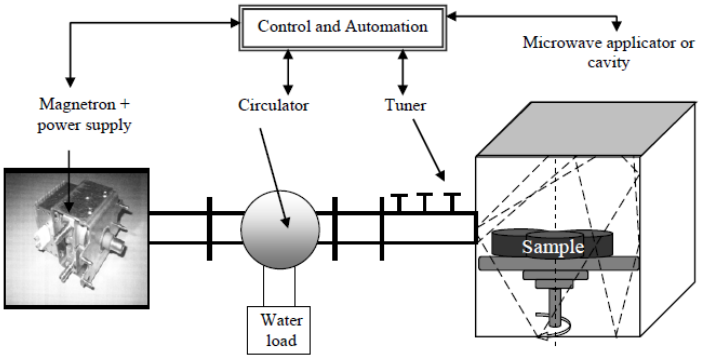


Fig.1 Configuration of the microwave curing package.

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.

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 5 to 70^{0} 2Θ at a rate of 2^{0} 2Θ per minute.

The compressive strengths of the concretes were tested using a compressive strength apparatus in accordance with the ASTM C39 [13] at 8 and 24 hours, and 7 and 28 days.

3. Results and discussion

3.1 Temperature rise and power histories

Fig. 2 shows the temperature increases and microwave power used throughout the curing time of the mortar (3CAM0.38_1:2.75) proportioned with Portland cement Type I: Ottawa sand at 1: 2.75 by weight and water-to-

cement mass ratio of 0.38. The temperatures increases are related to increases in of microwave power. This is because the heat generated by microwave energy varies in correspondence with the electric field intensity ($\vec{E}$), as shown in Eq. (1). In other words, when the microwave power increases, so too does the intensity of the electric field. Moreover, the temperature behaves similarly for all the monitored points. In terms of dielectric behavior, the SiO_2 in sand does not absorb microwave energy; therefore, the heat that accrues from microwave energy proceeds from hydration of the cement particles and water molecules.

$$Q=\sigma\left|\vec{E}\right|^2=2\pi f\varepsilon_0\varepsilon_r'\left(tan\,\delta\right)\left|E\right|^2 \quad (1)$$

Regarding the rate at which the temperature increases, in the first period during which microwave is introduced inwards to the microwave-cured mortar, the rate of increase is higher than that of the later period. This is because during the first period, the abundance of water inside the mortar consistently affects increases in the amount of heat generated. On the other hand, during the later period the water's composition changes to C–S–H and is evaporated outward, which causes a gradual reduction in the amount of heat generated as the amount of available water, especially free water, decreases.

It was found that the rate at which temperatures increases at the end of curing, corresponds to the power level of the microwave energy applied. For example, at the top surface where water evaporation take place, the final temperature at microwave power levels 180, 390, and 811 watt run at 69 ^{0}C, 123 ^{0}C, and 190 ^{0}C, respectively. This shows that though the microwave power level was increased up to two times, the temperature does not raise a half of the microwave power level. This high rate of heat generation at high power levels directly increases more rapidly the rate at which the temperature of the heated mortar increases. In addition, the temperature as immediately generated can accelerate hydration; that is, this reaction also generates heat.

Fig. 3 shows the temperature inside the specimen to be cured by microwave and power history during the application of microwave energy to a concrete (3CAC0.38_1:1:1) with different microwave power levels. The concrete is proportioned as follows: Portland cement:Ottawa sand:crushed lime stone rock equal to 1:1:1. This temperature behavior differs in clear ways from the temperature behavior observed in the mortar. In detail, during the early period of microwave curing the temperature increases rapidly; that is, it rises by an average of every 5 minutes. Subsequently, the temperature increases at a constant rate. All the monitored points—the top surface, bottom surface, and inside—behave similarly in this regard, which indicates that the presence of crushed lime stone rock affects the rate at which the temperature increases as well as the final temperature achieved. This may be because crushed limestone rock has a rough surface to which it is easier for water molecules to attach than the smoother surface of Ottawa sand [14]. This result shows that the heat generated within this sample is lower than that of is generated in the mortar sample. This means that when

microwave energy is introduced to the concrete sample, the sample maintains temperature or absorbs heat better than does the mortar under similar conditions.

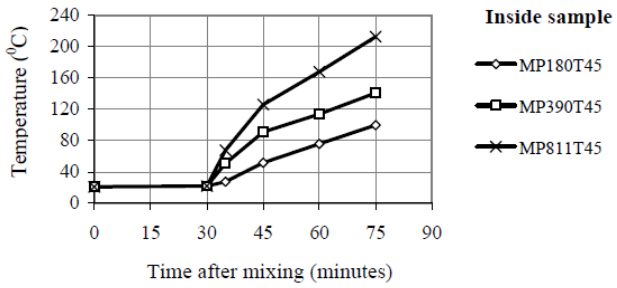


Fig. 2 Temperature and power history during the application of microwave energy to an inside of the mortar sample with different microwave power levels.

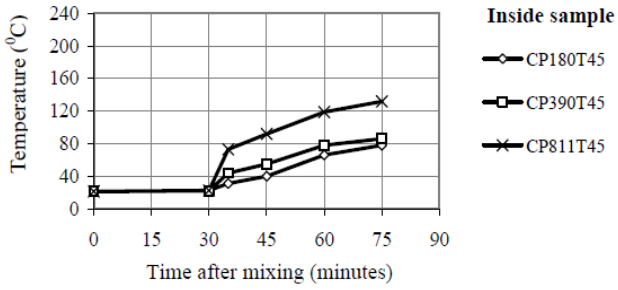
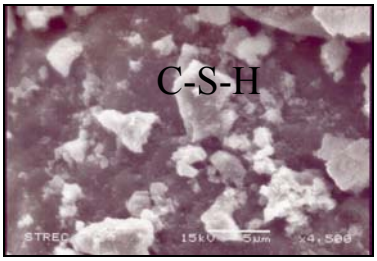


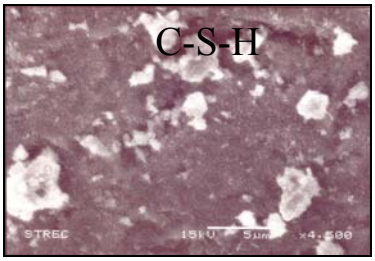
Fig. 3 Temperature and power history during the application of microwave energy to an inside of the mortar sample with different microwave power levels.

3.2 Morphology

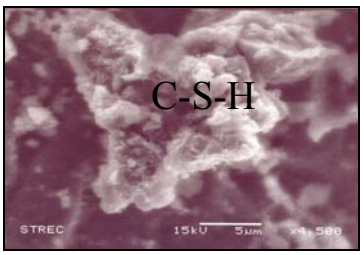
Typical micrographs of the mortar (3CAM0.38_1:2.75) subjected to microwave energy, and concrete (3CAC0.38_1:1:1) subjected to microwave energy at 180 watt for 45 minutes are shown in Fig. 4. It is not clearly seen from Fig. 4(a) and 4(f) that the samples consist of hydrated phases and pores. On its surface, the large particles appear to be agglomerated with a small particle, especially in the microwave-cured concrete.



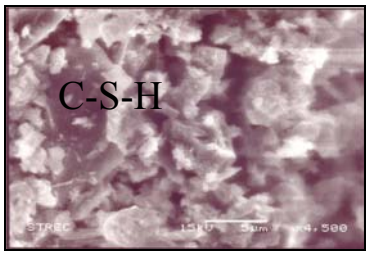
(a) 3CAM0.38_1:2.75 at a microwave power 180 watt



(b) 3CAC0.38_1:1:1 at a microwave power 180 watt



(c) 3CAM0.38_1:2.75 at a microwave power 390 watt



(d) 3CAC0.38_1:1:1 at a microwave power 390 watt

Fig. 4 Micrographs of mortar and concrete with different microwave power levels.

3.3 Atom ratios of Si/Ca versus Al/Ca

The EDX results for the cement pastes subjected to microwave energy are shown in Fig. 5. The atom ratio of Si/Ca versus Al/Ca for

the 0.38-w/c pastes after the application of microwave power of 390 watt for 45 minutes shows that for the normal paste, the Si/Ca cluster versus the Al/Ca cluster takes place in the narrow range of Si/Ca equal to 0.163–0.240, but with a wider range of Al/Ca equal to 0.022-0.090. In the mortar and concrete, the degrees of scatter for Si/Ca versus Al/Ca are greater than those of the other pastes. It is likely that the aggregate of the paste affects the distribution of the Si, Ca, and Al elements, causing them to become more widely dispersed.

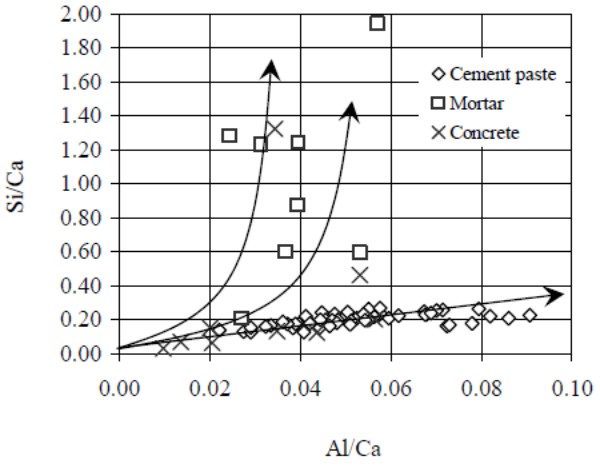


Fig. 5 Atom ratio of Si/Ca versus Al/Ca of cement paste, mortar and concrete after applying microwave energy with different power levels.

3.4 Phases

Figs. 6 and 7 show the X-ray patterns of the hydrated products in the mortar and concrete after microwave power of 390 watt had been applied for 45 minutes. For mortar, the phases identified include hatrurite (Ca_3SiO_5) and lanite (Ca_2SiO_4). Similar to mortar, the concrete consists mainly of hatrurite (Ca_3SiO_5) and quartz (SiO_2).

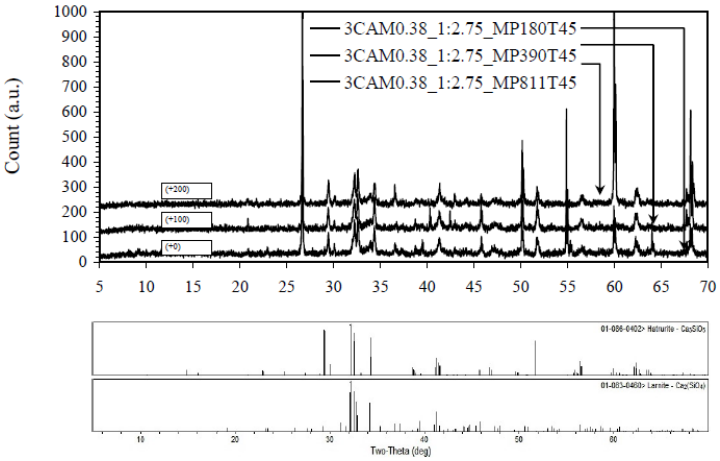


Fig. 6 X-ray diffraction of mortar after applying microwave energy with different power levels.

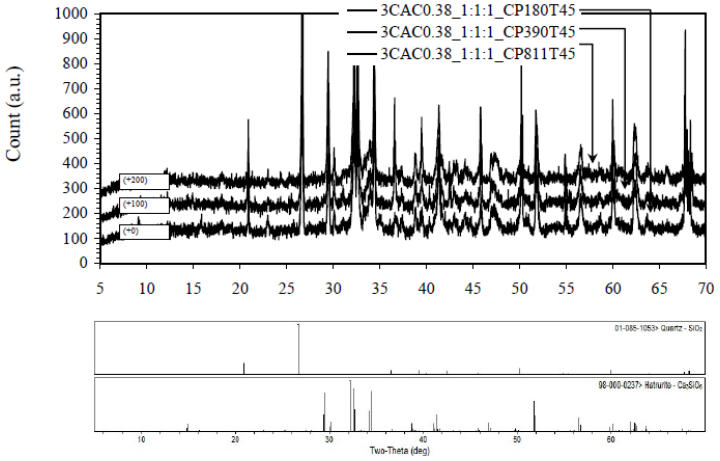


Fig. 7 X-ray diffraction of concrete after applying microwave energy with different power levels.

3.5 Compressive strengths

The compressive strengths of the mortar of (3CAM0.38_1:2.75) when subjected to microwave energy, and concrete (3CAC0.38_1:1:1) compared to normal curing are shown in Fig. 8, when cured at elevated temperatures using an microwave power level of 180 watt, mortar can develop strength quite rapidly, and with a higher microwave power level the development of strength decreases. It could be that an increase in microwave power significantly affects the interfacial transition zone between the neat paste and the aggregate [15].

From the literature, increases in temperature expand the range of the interfacial transition zone and as a consequence compressive strength is reduced.

Similar to the mortar case, the microwave-cured concrete corresponds to the microwave power levels shown in Fig. 9, the compressive strength of concrete decreases with increases in the power level, as previously discussed earlier.

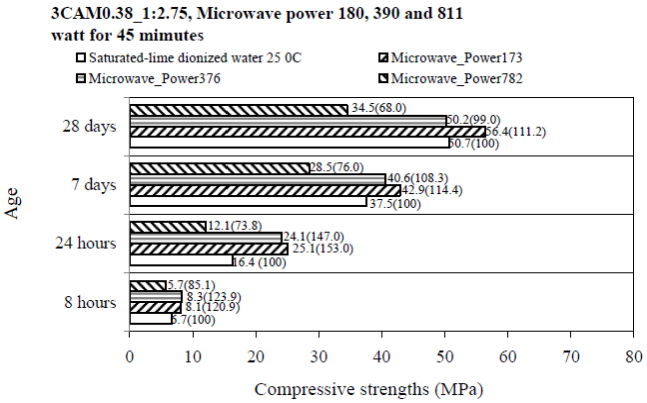


Fig. 8 Compressive strengths of mortar after applying microwave energy with different power levels.

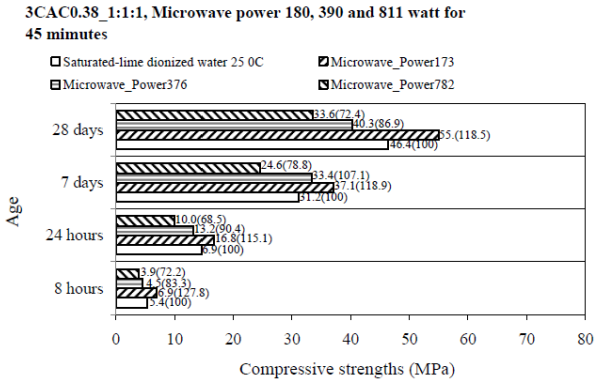


Fig. 9 Compressive strengths of concrete after applying microwave energy with different power levels.

4. Conclusions

The temperature increased monotonically among the positions of measurement during the microwave-curing process. The typical micrographs of the microwave-cured concretes at the age of 4 hours 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.

For compressive strengths, the microwave-cured concretes developed strength quite rapidly in accord with the maintenance of the level of microwave power and time of application. At the age of 8 hours after microwave curing at a power 180 watt, 0.38-w/c concrete attained a strength of 6.90 MPa (127.8% higher than the lime-saturated water-cured concrete).

5. Acknowledgement

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