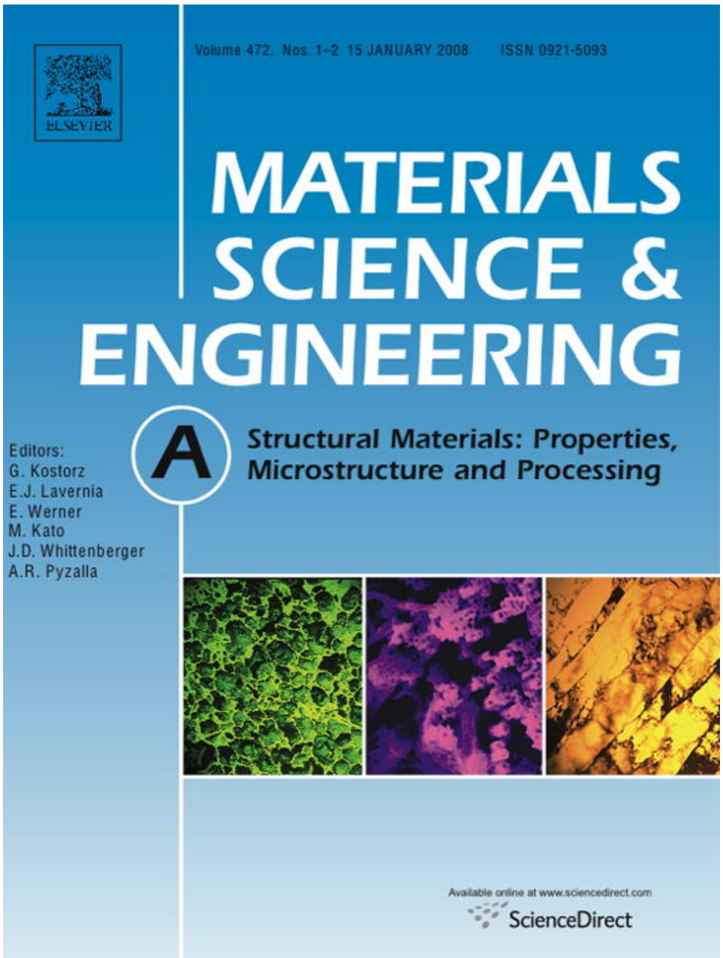
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Development of compressive strength of cement paste under accelerated curing by using a continuous microwave thermal processor

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Abstract

A high rate of strength development in mortar and concrete can benefit a number of important operations in the construction industry, such as concrete precasting, pavement repair and concrete decontamination. In this study, the acceleration of cement paste curing with microwave energy by using continuous belt drier is investigated. The microwave power was generated by means of 14 compressed air-cooled magnetrons of 800 W each for a maximum of 11.2 kW. The power setting could be adjusted individually in 800 W steps. This study included the heat transfer analysis taking place during the curing of cement paste with microwave energy and the compressive strength development of cement paste. The tested results were compared with those of the conventional cement paste that were cured in water and air. Internal structures of cement paste were investigated for analyzing the mechanical properties after curing. The variables emphasized on the thermal influences from using microwave energy, properties of cement paste, and time of curing. The test results showed that microwave energy accelerated the early-aged compressive strength of cement paste and not affect upon later-aged strength; for instance, the growth rate of compressive strength of 30 min-cured and water-to-cement ratio of 0.4 microwave-cured mortar after 3 days was 103% while 101 and 95% for specimens at the ages of 7 and 28 days, respectively. Furthermore, microwave curing can be saved profitably on energy consumption and reduced time of curing.

Keywords: Compressive strength; Cement paste; Microwave; Heat transfer

1. Introduction

During the past decade, there are many successful examples of microwave application including; the heating and drying of foods, heating and drying of ceramics, heating and drying of concrete and vulcanizations of rubber. The microwave heating process takes place inside the material, the penetrated depth of which governs how strongly the microwaves are absorbed. It is known that the heat dissipated from the microwave energy depends on many parameters such as the configuration and geometry of the dielectric samples, the microwave power level, the microwave field distribution and the dielectric properties of dielectric samples. A number of analyses of the microwave heating process have appeared in recent literature [1–29]. An excellent review of the drying techniques in dielectric materials using microwave energy has been presented by Mujumdar [1], Metaxas and Meridith [2] and Schubert and Regier [3].

The use of alternative techniques for heating cement paste and concrete has been investigated sporadically in the last 20 years. Current heat curing practice in cement past or concrete products is generally based on the application of conductive heat through either direct contact with steam, hot water-/steam-heated casting beds or in high-pressure steam autoclaves. The drive for production efficiency and increasing awareness of the environmental impacts of manufacturing has prompted research into alternative methods for rapid curing of concrete. There have been relatively few new innovations in rapid heat curing technologies over the last few decades. In recent years, the use of industrial microwave heating technology for rapid curing of concrete has been investigated and developed continuously by many researchers at both lab scale and pilot scale [25–30]. Microwave heating is based on the internal energy dissipation associated with the excitation

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of molecular dipoles and ions in electromagnetic fields. The use of microwave heating to process concrete potentially covers the entire spectrum of:

- preheating of materials prior to the product casting or primary curing stage;
- rapid curing and/or drying as a primary process stage because microwave energy can accelerate the hydration reaction of hydraulic cement, resulting in rapid strength development of concrete in an early period;
- drying as a secondary post-curing stage.

From practical point of view, it is well known that for a given microwave system, a load (sample) placed in different locations inside the applicator absorbs microwave differently. Furthermore, despite the high number of stimulated modes, often a non-uniform field distribution that is constant in time will develop. This field distribution depends mainly on the cavity size, the product geometry and the dielectric properties of the material to be processed. In contrast to single-mode application, normally this non-uniform field distribution, which would result in non-uniform heating pattern, is not desired, since it is difficult to control. An undesired non-uniform heating pattern can be prevented by changing the field configuration either by varying cavity geometries (e.g., mode stirrer) or by moving the product (on a conveyer belt or turntable) [3].

It is expected that the continuous microwave applicators technique has possibly introduced soon. The reason for this is that energy conservation, labour savings and rationalization of production are seriously promoted in the high energy consuming ceramic and rubber industries. Particularly, drying and forming processes in the curing of concrete products need a lot of time and energy, which results in a lack of rationality. Only the introduction of some continuous production system through the efficient use of a rational energy source can solve this problem.

Although a number of studies have been conducted on simple microwave heating process. Most of them were carried out using a domestic or housing microwave oven and a single or multimode cavity with a non-movable material. Those studies show that the result may be dependent on the method used to carry out the curing or heating process. However, numerical simulation and experimental work of heat curing practice in cement past, mortar or concrete by a continuous microwave belt drier have not been systematically reported.

The objective of this study is to demonstrate the applicability of microwave energy, as an energy-saving and production-cost-reducing technology. The accelerated curing method using the microwave heating technique for later-age strength development is illustrated. The study is the microwave curing of cement paste in a continuous microwave belt drier where a series of 14 magnetrons of 800 W each with total power of 11.2 kW were installed. The experimental results from this study could help to identify some of these potential problems during the practical design stage. This study is of great importance from the practical point of view because it shows the possibility of application of microwave heating-drying of dielectric materials on an industrial scale, especially in a continuous system.

2. Experimental procedure

Microwave heating was carried out in a microwave continuous belt drier (Fig. 1). The microwave cavity was cylindrical shape with exterior dimensions of 5300 mm × 1200 mm × 1600 mm. The drier operates at a frequency of 2.45 GHz with maximum working temperature of 230 °C. The microwave power was generated by means of 14 compressed air-cooled magnetrons of 800 W each for a maximum of 11.2 kW. The power setting could be adjusted individually in 800 W steps. In the continuous processing equipment, two open ends were essential, through which the material to be heated up on the belt conveyer is put in and taken out. In this equipment, leakage of microwaves is prevented by the countermeasure in double with a combination of mechanical blocking filter and microwave absorber zone filter to be provided each at the open ends. The microwave leakage was controlled below the DHHS (US Department of Health and Human Services) standard of 5 mW/cm^2. The multiple magnetrons were installed in an asymmetrical position around the rectangular cavity (Fig. 1). The microwave power

Fig. 1. (a) Continuous microwave belt drier for drying tableware product, (b) location of 14 magnetrons.

Table 1
Mix proportions of cement paste used

Water-to-cement ratio (w/c)	Portland cement type I (kg/m^3)	Water (kg/m^3)
0.30	1620	486
0.35	1498	524
0.40	1394	558
0.45	1303	586
0.50	1223	612

was then directed into the drier by means of waveguides. An infrared camera (located at the at the opening ends) was used to measure the temperature of the samples (accurate to ±0.5 °C). The cement paste specimens were made with dimensions of 5 cm × 5 cm × 5 cm. By mixing Portland cement type I which had a Blaine Fineness of 3250 cm^2/g together with tap water (pH 7.0) as following the ASTM C305 standard [31], in addition, the water-to-cement ratios (w/c) were kept constantly of five cases (0.30, 0.35, 0.40, 0.45, and 0.50). The mix proportions of cement paste are shown in Table 1. After mixing and placing the cement paste slurry into mould, plastic sheet was used to cover for protecting moisture loss. At age of 23.5 ± 0.5 h after mixing, the specimens were demolded and cured in different methods, including microwave cured, water cured, and air cured. Under water curing method, 15 specimens are soaked in water that were controlled temperature of 25.0 ± 2.0 °C until testing time. In addition, 15 specimens were cured in under both of temperature and relative moisture of 25.0 ± 2.0 °C and 60.0 ± 5.0%, respectively. The compressive strength of cement paste was tested at the ages of 3, 7, 14, and 28 days as following the ASTM C39 [31]. While 15 of the samples were microwave cured. The microwave-cured samples were weighed before and after they were transferred to the microwave cavity via belt conveyer. The samples to be dried passed through the drier on an air-permeable microwave transparent conveyor belt. After a certain time, microwave power was applied for a specified period of time (15 and 30 min). During the heating process, microwaves penetrated the sample, heating the water until it diffused to the surface. The humid air was then drawn out of the cavity by a suction system. While the curing process proceeded, temperature variations of specimens were measured by using infrared camera for 60 min from the curing beginning. The dried samples left the cavity through another opening for further characterization. In this stage, the 15 samples were then removed from the cavity and weighted again to determine the moisture loss. The moisture content of microwave-cured specimen can be calculated based on dry basis from the weight loss of cement paste specimens before and after microwave curing comparing to weight after curing one. A plastic sheet was wrapped around the samples to prevent further moisture loss. For the three samples tested at 1 day, they were removed from the cavity after 3 h and capped for compression testing. The other 12 microwave-cured samples were also removed from the cavity after 3 h and cured in air room (controlling temperature and humidity) for 3, 7, 14 and 28 days, respectively, until they are tested. The 30 conventional cured samples are covered with plastic sheets after casting, removed from the mould after 1 day, and cured in water bath (15 samples) and air room (15 samples) and tested at the ages of 1, 3, 7, 14 and 28 days, respectively, to establish a reference strength against with microwave-cured samples can be compared.

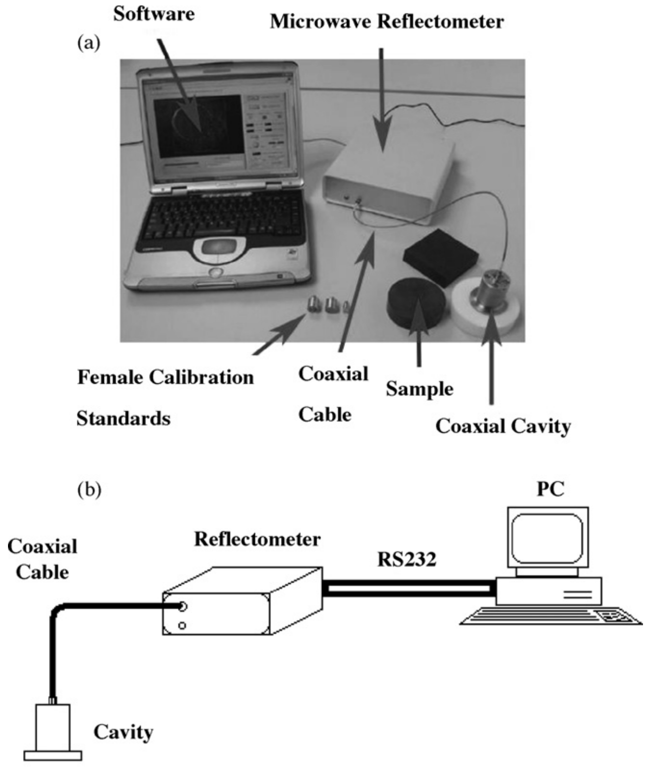


Fig. 2. Portable dielectric measurement (Network Analyzer).

The dielectric properties for cement paste samples were measured at 25 °C using a portable dielectric measurement (Network Analyzer) over a frequency band of 1.5–2.6 GHz as shown in Fig. 2. The portable dielectric measurement kit allows for measurements of the complex permittivity over a wide range of solid, semi-solid, granular and liquid materials. It performs all of the necessary control functions, treatment of the microwave signals, calculation, data processing, and results representation. The software controls the microwave reflectometer to measure the complex reflection coefficient of the material under test (MUT). Then it detects the cavity resonant frequency and quality factor and converts the information into the complex permittivity of the MUT. Finally, the measurement results are displayed in a variety of graphical formats, or saved to disk.

In this study, various main parameters, including microwave power level, water-to-cement ratio (w/c) and heating time were studied using cement paste. The heating characteristics were also studied.

3. Microwave and dielectric material interactions

In convective drying, dry air is used to take away surface water saturation from the dried sample, therefore creating a pressure gradient between the surface and inner part, which causes moisture migration from inside the sample to the surface. In this process, the temperature gradient will enhance the ability of dry air to remove water from the surface and increase the moisture migration rate within the sample. But there are many disadvantages with this method. Among these are low energy efficiency and lengthy drying time during the falling rate period. This is mainly caused by rapid reduction of surface moisture and consequent shrinkage, which often results in reduced heat transfer. In contrast to conventional drying, microwave heating is volumetric and non-uniform. Samples dried less than a few skin depths in thickness. The resonance effect can occur inside the material which results in the field distribution not having an exponential decay from the surface. In some cases the highest field strength, and therefore power density, can actually occur in the center of the sample. This is caused by the interference of waves reflected from the back side of sample. This mechanism pushes moisture out of product with great efficiency, as the moisture content of the product decreases.

Industrial applications mostly need continuous processing due to the high throughputs desired. Therefore, continuous microwave applicators have been developed. Today's microwave system in industrial applications may be divided into two groups by the number and power of microwave sources: high-power single magnetron and low-power multi-magnetron devices (discussed in this work). Whereas, for a single-mode unit only a single source is possible, in all other systems, the microwave energy can be irradiated optionally by one high-power magnetron or several low-power magnetrons. Whereas, common industrial high-power magnetrons have longer operating lifetimes, low-power magnetrons have the advantage of the very low prices, due to the high production numbers for the domestic market [3].

In analysis of dielectric heating, the root-mean-square value of the electric field intensity E is normally used to evaluate the microwave energy absorbed. Thus, the microwave energy absorbed, or local volumetric heat generation term can be defined as [20]:

$$Q = \sigma |E|^2 = 2\pi f \varepsilon_0 \varepsilon_r' (\tan\delta) E^2 \qquad (1)$$

where Q is the microwave energy absorbed, σ the effective conductivity, f the frequency, ε_0 the permittivity of free space (8.86×10^{-12} F/m), ε_r' the relative dielectric constant, $\tan\delta$ the loss tangent coefficient and E is the electric field intensity.

In above equation, the microwave energy absorbed is proportional to the frequency of the applied electric field and dielectric loss factor, and is proportional to the square of local electric field. This equation is crucial in determining how a dielectric material will absorb microwave energy when it is placed in a high frequency electric field. However, the interaction between electromagnetic field and dielectric material during applied microwave field which is depending on the dielectric properties of that materials.

When the material is heated unilaterally, it is found that as the dielectric constant and loss tangent coefficient vary, the penetration depth will be changed and the electric field within the dielectric material is altered. The penetration depth is used to denote the depth at which the power density has decreased to 37 % of its initial value at the surface and can be defined as [22].

$$D_p = \frac{1}{(2\pi f/\upsilon)\sqrt{\varepsilon_r'\left(\sqrt{1+(\varepsilon_r''/\varepsilon_r')^2}-1\right)/2}} = \frac{1}{(2\pi f/\upsilon)\sqrt{\varepsilon_r'\left(\sqrt{1+(\tan\delta)^2}-1\right)/2}} \qquad (2)$$

where D_p is the penetration depth, ε_r'' the relative dielectric loss factor and υ is the microwave speed.

The penetration depth of the microwave power is calculated according to Eq. (2), which shows how it depends on the dielectric properties of the material. It is noted that products with huge dimensions and high loss factors, may occasionally overheat a considerably thick layer on the outer layer. To prevent such phenomenon, the power density must be chosen so that enough time is provided for the essential heat exchange between boundary and core. If the thickness of the material is less than the penetration depth, only a fraction of the supplied energy will become absorbed.

Furthermore, consider the dielectric properties of cement paste cement paste and concrete samples typically show moderate lossiness depending on the actual composition of the material. With large amount of cement content typically shows slightly greater potential for absorbing microwaves. For all cement paste samples, a decrease in the water/cement ratio typically increases ε'', accompanied by a slight reduction in D_p.

4. Results and discussions

Figs. 3–5 show the temperature variations with respect to elapsed times for different testing conditions. It is found that at a high microwave power level the temperature profile of the samples continuously rises faster than that in the case of low microwave power level. The influence of w/c ratios on temperature profiles is also depicted in Figs. 3–5. It is observed that an increase of w/c ratios from 0.3 to 0.5 gives very different temperature profiles. The large amount of cement content (or lower w/c ratio) typically shows a greater potential for absorb-

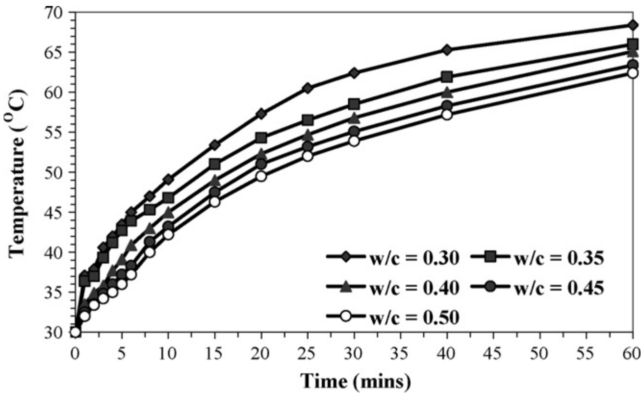


Fig. 3. Temperature variations vs. elapsed times for different w/c ratios of cement paste (1 magnetron).

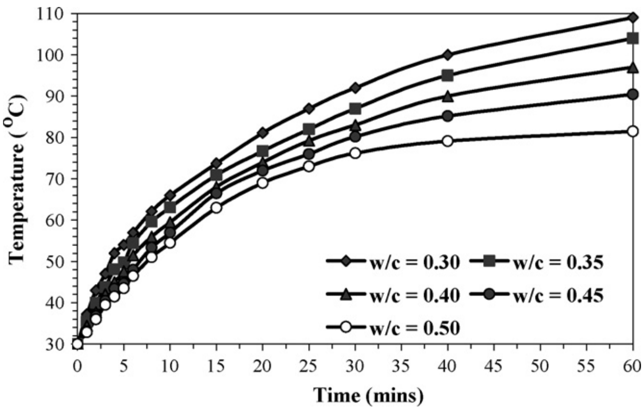


Fig. 4. Temperature variations vs. elapsed times for different w/c ratios of cement paste (2 magnetrons).

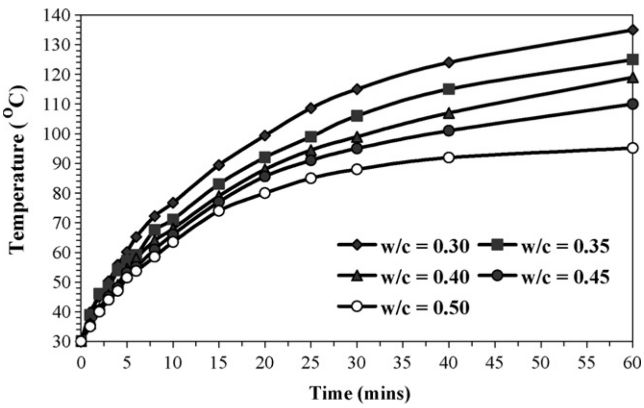


Fig. 5. Temperature variations vs. elapsed times for different w/c ratios of cement paste (3 magnetrons).

ing microwaves. In addition, for samples with a lower w/c ratio, there is higher heat liberated from the hydration reaction. This phenomenon explains why the temperature profiles in case of lower w/c ratio are higher than those in the case of higher w/c ratio. As shown in Fig. 6 which has redrawn from Figs. 3–5, the temperature profile of samples rises faster in the case of high microwave power level.

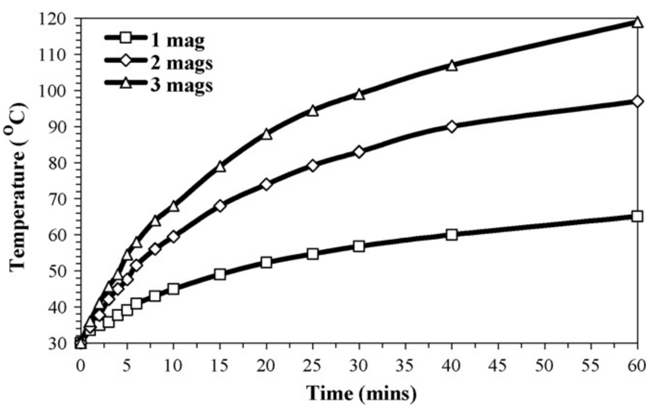


Fig. 6. Temperature variations vs. elapsed times for different microwave power levels (numbers of magnetrons) (at w/c = 0.4).

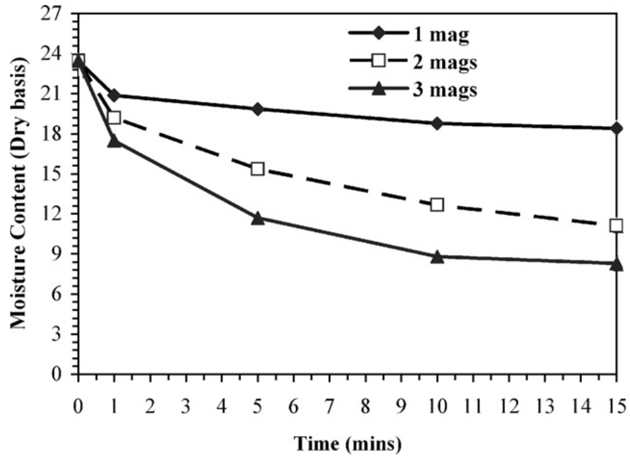


Fig. 7. Moisture profile vs. elapsed times for different microwave power levels (numbers of magnetrons) (at w/c = 0.4).

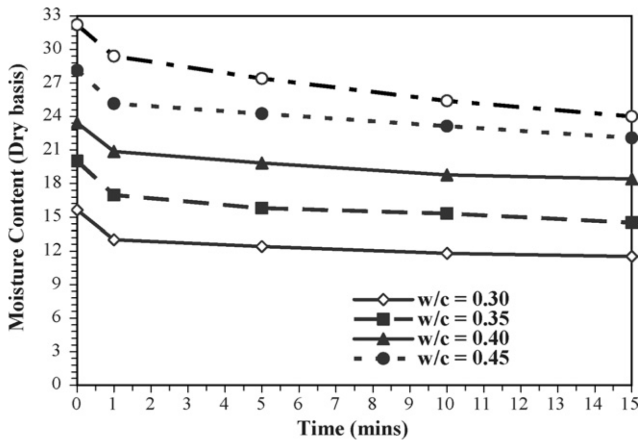


Fig. 8. Moisture profile vs. elapsed times for different w/c ratios of cement paste (1 magnetron).

Fig. 7 shows moisture profiles with respect to elapsed times for different microwave power levels with constant w/c ratio. It is found that at a high microwave power level the moisture profile of the samples continuously decreases faster than that in the case of low microwave power level. This phenomenon corresponds to the level of absorbed energy in samples as explained above.

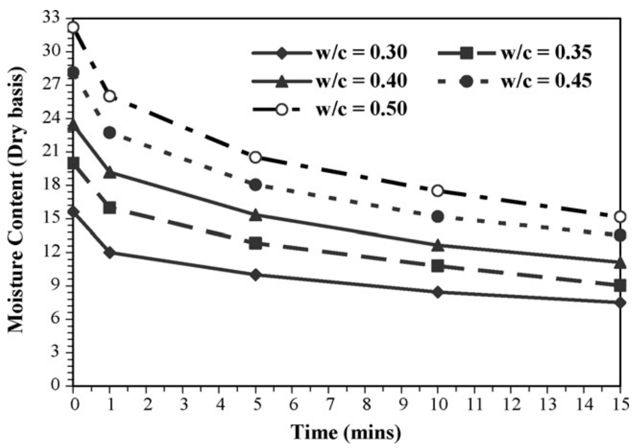


Fig. 9. Moisture profile vs. elapsed times for different w/c ratios of cement paste (2 magnetrons).

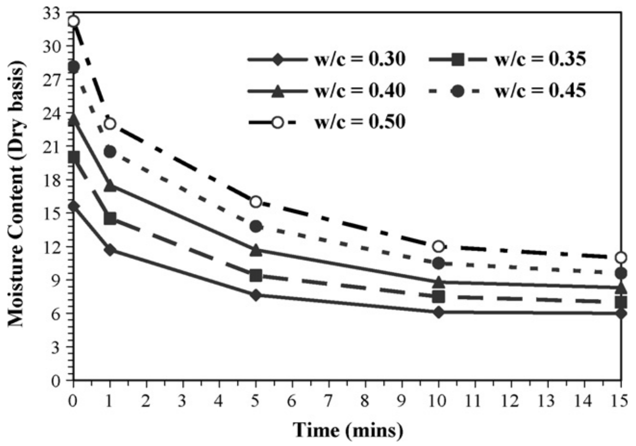


Fig. 10. Moisture profile vs. elapsed times for different w/c ratios of cement paste (3 magnetrons).

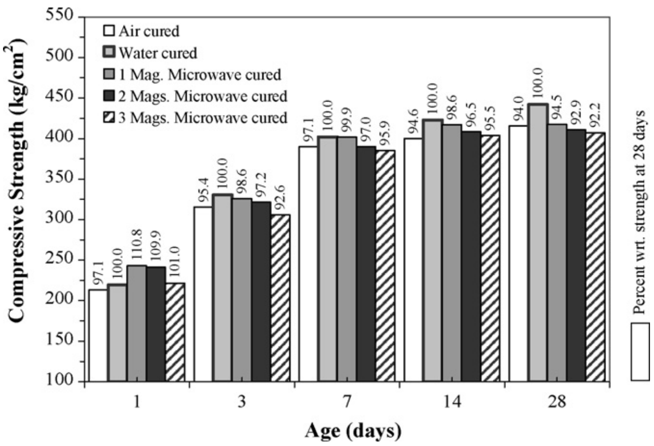


Fig. 12. Comparison of compressive strength development for different curing methods (w/c = 0.4 and 15 min microwave cured).

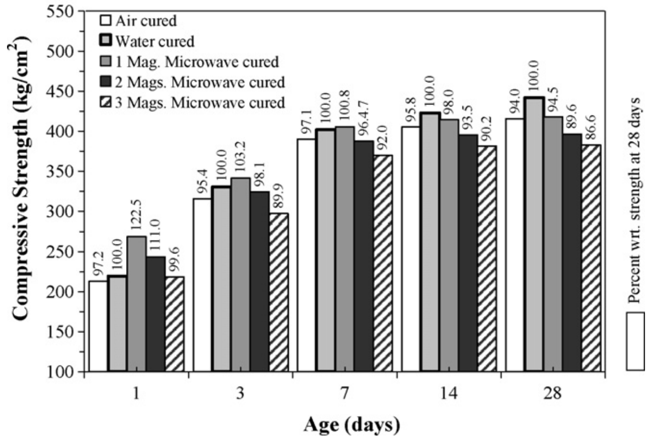


Fig. 13. Comparison of compressive strength development for different curing methods (w/c = 0.4 and 30 min microwave cured).

Figs. 8–10 show the moisture profiles with respect to elapsed times for different testing conditions. It is found that at a high microwave power level the moisture profile continuously drops faster than that in the case of low microwave power level. The influence of w/c ratios on moisture profiles is also depicted in Figs. 8–10. It is observed that an increase of w/c ratios from 0.3 to 0.5 gives very different moisture profiles. The lower w/c ratio corresponding to higher dielectric loss factor typically shows a greater potential for absorbing microwaves. It is evident from the figure that the sample dries quickly throughout. In particular, the bulk of this sample that receives the largest amount of microwave energy absorbed. It would correlate to microwave energy absorbed which depends on the changing of the configuration of electromagnetic field in the sample due to the variation of moisture content [19]. Furthermore, near the end stage of curing process as the moisture content inside the sample is reduced, this decreases the microwave energy absorbed. Thus, equilibrium is reached between microwave drying and convective losses by lowering sample temperature.

Figs. 11–15 show the strength of cement paste sample presented in term of compressive strength after curing with different conditions where the several samples were tested. It can be seen from Fig. 11 that microwave-cured samples at different w/c ratios with constant microwave power level (1 magnetron) give very different compressive strength profiles. The compressive strengths of all w/c ratios increase quickly at early stage

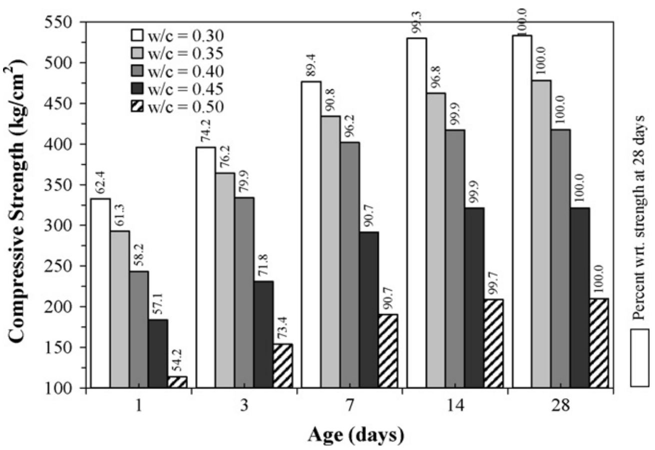


Fig. 11. Compressive strength development for different w/c ratios for cement paste specimens (1 magnetron and 15 min microwave cured).

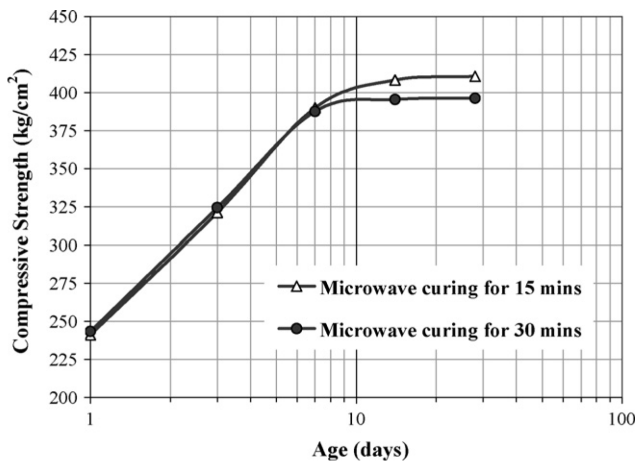


Fig. 14. Compressive strength development for different microwave curing time (w/c = 0.4 and 2 magnetrons).

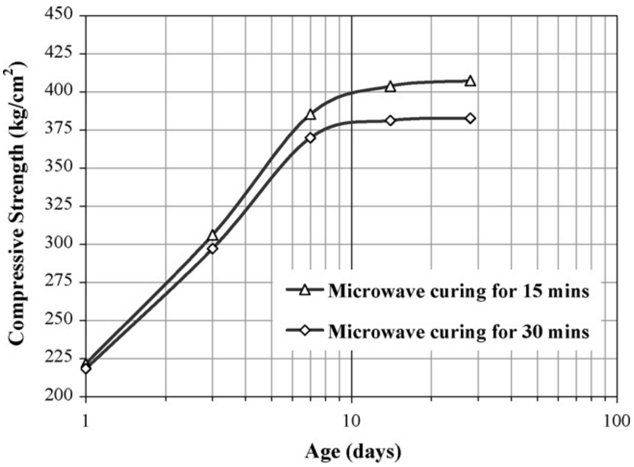


Fig. 15. Compressive strength development for different microwave curing time (w/c = 0.4 and 3 magnetrons).

until 7 days. After 7 days, the strength of all cases appears to reach a plateau. It is observed that the strengths of microwave cured at lower w/c ratio are higher than that for microwave cured at higher w/c ratio. This can be explained by higher microwave energy absorbed reached by case of lower w/c ratio in the curing process. From Figs. 12 and 13, for cement paste samples, a w/c ratio of 0.4 is employed. Several important observations can be made. The compressive strengths of both microwave-cured and standard-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 samples and control sample attain similar degree of hydration. After 7 days, the strength appears to reach a plateau while at the long term the strengths of microwave-cured samples are slightly lower than that for normal cured samples. Furthermore, for the cement paste samples subjected to higher power levels, which represent microwave curing with higher heating rates, therefore, the microcracks and pores is increased and the compressive strength is reduced. As shown in Figs. 14 and 15 which were redrawn from Figs. 12 and 13, the compressive strength of both 15 min microwave cured and 30 min microwave cured is almost identical at early stage until 7 days. After 7 days, the strength appears to reach a plateau while at the long term the strengths of 15 min microwave cured are slightly higher than that for 30 min microwave cured. This is because microwave cured at short time exhibits less microcracks corresponding to a better microstructure arrangement and the compressive strength is improved.

It can be concluded from the tested results that microwave energy can be not only accelerated the early compressive strength of cement paste without affecting strength at the age of 28 days but also saved energy and reduced time of curing.

The following steps, the internal structures of cement paste are investigated for analyzing the mechanical properties after curing. Fig. 16 shows the texture overview of the cement paste samples under various cured processes by using scanning electron microscope (SEM) technique. It is found that the cured samples in all cases seem to have a similar microstructure arrangement. The results, while preliminary, confirm that the microwave curing method can potentially produce cement paste with very high early strength and little deterioration in its long term performance. In this investigation, the possibility of developing very high early strength without significantly affecting the long term strength is illustrated.

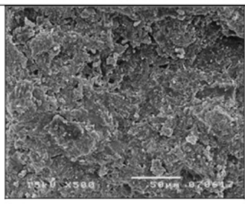
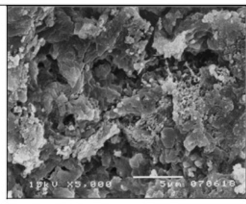
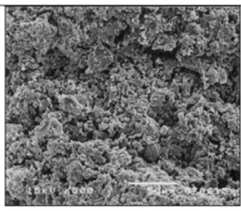
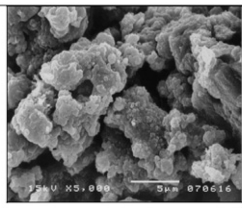
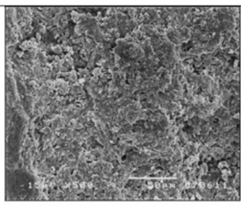
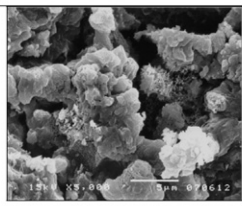
Curing method	500 Magnifications	5000 Magnifications
Water cured		
Air cured		
Microwave cured		

Fig. 16. Microstructure of cement paste after curing in different methods.

In addition, considering thermal runaway effect, it is well known that the temperature dependence of the dielectric properties varies according to the material in question and is often very complex. The values of these properties may increase with temperature of decrease with temperature. At room temperature, the cement paste, which exhibits a rapid increase in the loss factor with increasing temperature. In such cases, when this phenomenon known as thermal runaway is apparent, damage such as poor product quality and sample cracking may occur to the product being heated. In this study, great care must be taken to monitor temperatures and turn off the microwave power before the danger period commences. In some instances when this phenomenon cannot be controlled, especially for materials where the moisture content is close to bone dry or where dried out areas may occur in the product, microwave heating may not be feasible. Understanding, prediction, and preventing or controlling thermal runaway present a major challenge to the development of microwave processing.

As mentioned in the previous work, especially by the authors [22], indicated obviously that the energy consumption by using microwave energy can save potentially more than the conventional curing. Nevertheless, in this primary study aimed on curing of cement paste which has lack of essential factors for assessment the energy consumption and cement paste has used in specific purpose in structural works such as grouting, whereas concrete is produced in general purpose; therefore, estimate on energy consumption in concrete is suitable to perform.

5. Conclusions

Microwave energy can accelerate the hydration of cement paste, resulting in rapid strength development of cement paste in an early period. Microwave heating using a continuous belt drier provides relatively deeper penetration and displays an uniformity heated pattern when compared to that achieved using other simply microwave heating systems as well as conventional steam heating. The deeper penetration achieved with microwave heating also means that, unlike conductive heating with steam, compressive strength and near-surface quality are not adversely affected when bulk heating rates are doubled, in example, from 20 to 40 °C/h.

The tested results indicated that microwave energy can be not affect strength development of cement paste; for instance, the growth rate of compressive strength w/c = 0.4 microwave-cured mortar that was used 1 magnetron applied and 30 min-cured and after 3 days was 103%, while 101 and 95% for specimens at the ages of 7 and 28 days, respectively. Our results, while preliminary, show that the microwave curing method can potentially produce cement paste with very high early strength and little deterioration in its long term performance.

Furthermore, by overall when handling a microwave continuous belt drier correctly, we can conclude that it will realize the following advantages, compared to other heating systems:

(1) faster, reproducible and more homogeneous heating (high product quality);
(2) faster heating of thicker layers because the multiple magnetrons are arranged around the cavity, the microwaves can penetrate further into the multi-plane of material;
(3) microwave energy can accelerate the hydration of cement, resulting in rapid strength development of concrete in an early period;
(4) immediately ready for operation and control of heat capacity without delay;
(5) no heat storage losses;
(6) low specific energy consumption.

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