Original Research

# Hydration and Microwave Curing Temperature Interactions of Repair Mortars

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## Abstract

Microwave curing of repair patches provides an energy efficient technique for rapid concrete repair. It has serious economic potential due to time and energy saving especially for repairs in cold weather which can cause work stoppages. However, the high temperatures resulting from the combination of microwave exposure and accelerated hydration of cementitious repair materials need to be investigated to prevent potential durability problems in concrete patch repairs. This paper investigates the time and magnitude of the peak hydration temperature during microwave curing (MC) of six cement based concrete repair materials and a CEM II mortar. Repair material specimens were microwave cured to a surface temperature of 40-45 °C while their internal and surface temperatures were monitored. Their internal temperature was further monitored up to 24 hours in order to determine the effect of microwave curing on the heat of hydration. The results show that a short period of early age microwave curing increases the hydration temperature and brings forward the peak heat of hydration time relative to the control specimens which are continuously exposed to ambient

conditions (20 °C, 60% RH). The peak heat of hydration of normal density, rapid hardening Portland cement based repair materials with either pfa or polymer addition almost merges with the end of microwave curing period. Similarly, lightweight polymer modified repair materials also develop heat of hydration rapidly which almost merges with the end of microwave curing period. The peak heat of hydration of normal density ordinary Portland cement based repair materials, with and without polymer addition, occurs during the post microwave curing period. The sum of the microwave curing and heat of hydration temperatures can easily exceed the limit of about 70 °C in some materials at very early age, which can cause durability problems.

**Keywords**
Microwave curing; concrete repair; heat of hydration

## 1. Introduction

The temperature of cementitious materials when mixed with water and their subsequent exposure to the environmental conditions has a significant effect on the hydration process. The hydration process slows down at low temperature and may stop in freezing conditions. Higher temperature leads to an increase in the reaction rate according to Arrhenius law and curing temperature plays an important role in the strength development and microstructure formation of cementitious materials [1, 2]. In practice, concrete repair patches can be exposed to different ambient temperatures, deliberately applied external heat during curing in addition to the heat of hydration. Copeland and Kantro [3] reported an increase in the initial rate of hydration of alite and belite at higher temperature. Kjellsen and Detwiler [4] carried out an experimental investigation on the hydration reaction kinetics of Portland cement mortar cured at temperatures increasing incrementally from 5 °C to 50 °C. They reported an increasing degree of hydration for higher temperature applied at early age. A study carried out by Escalante-Garcia and Sharp [2] showed that higher initial temperature of OPC accelerates hydration and, therefore, results in higher heat evolution. Hydration temperature of cementitious repair materials has a strong influence on the strength and durability of a repair patch in the short and long term [5, 6].

Thermal curing methods such as steam curing or autoclaving are commonly used in precast concrete practice to accelerate the curing of cementitious materials and hence achieve a higher early age compressive strength. Gallucci et al. [7] reported that C-S-H is highly sensitive to curing temperature which results in a continuous increase in apparent density of cement paste with curing temperature in the range of 5 to 60 °C. Traditional thermal curing methods often require 1 to 3 days of maintaining high temperature for strength development of concrete. Thermal curing methods which provide internal heat, such as electric and microwave curing, are now becoming important in order to accelerate concrete curing to shorter periods. The popularity of these approaches is due to the fact that they provide uniform heating and often require less than one hour of thermal curing. Most previous research on microwave curing deals with rapid high strength development of conventional concrete, usually focussed on the precast industry [8-11]. These studies have mainly focused on the effect of temperature at the end of the microwave curing period on strength

development without considering the heat of hydration. However, potential durability problems such as delayed ettringite formation and thermal shrinkage cracking can occur due to the high temperature reached at early age under microwave curing [12]. These temperatures and the durability concerns can be further increased by the interaction of heat of hydration at early age with microwave curing temperature. This important aspect which is addressed in this investigation has so far received little attention.

Very little information is available in literature on the combined effect of heat generated by microwave curing and hydration temperature on conventional concrete made with ordinary Portland cement. This lack of knowledge is even greater for concrete repair materials and repair patches. Concrete repair materials used in practice are usually proprietary commercial formulations which incorporate various admixtures, cement replacement materials and polymers. These additives influence the exothermic chemical reactions of hydration which control the properties of the fresh and hardened mortar. For example, the different types of cement, admixtures and cement replacement materials used in self compacting concrete can significantly affect both the rate and magnitude of heat generated under normal curing conditions [13]. The heat of hydration of different concrete repair materials varies considerably and its combined effect with microwave curing temperature can lead to very high curing temperatures at early age especially in rapid setting repairs.

Much of the research reported on microwave curing of concrete assumes a uniform distribution of heat in the test samples [14]. Conclusions are often based on temperatures monitored on the top surface of an element or internally using thermocouples. The former method of monitoring is suitable for use on site to provide temperature control to an automated microwave curing system [15, 16]. The latter temperature monitoring method is possible in laboratory studies [8, 17]. Some theoretical studies show a decrease in microwave induced temperature as depth of the heated object increases [18, 19].

This research investigation reports the relationship between test results of internal and surface temperatures monitored on specimens of concrete repair mortars. The authors have previously reported on the parameters of microwave curing which affect the strength and other properties of concrete repair materials [20] including bond between a repair patch and both the substrate concrete and the steel reinforcement [21, 22]. However, the interaction between microwave curing and hydration temperatures at early age can have significant effect on the properties and long term deterioration processes in concrete repair patches [23]. There is a lack of information on the combined microwave curing and hydration temperatures developed in cementitious materials and their effect on properties including early age shrinkage, moisture loss and durability of concrete repairs. The current study investigates the heat of hydration for different proprietary cementitious repair materials when they are microwave cured to a temperature of 40-45 °C for up to 50 minutes to ensure that the combined total with early hydration temperature does not exceed 70º C. The 70ºC temperature limit is used for early age thermal curing of precast concrete to prevent durability problems caused by delayed ettringite formation [24]. It is a suitable target to adopt for microwave cured repair materials until future research provides a more precise limit based on durability studies of repair patches made with compositions representing proprietary repair materials.

This research paper represents part of a larger research project funded by the European Commission on microwave curing of concrete patch repairs and on the development of a prototype system for in-situ microwave curing of concrete patch repairs (FP7 MCure project). The prototype

has been successfully tested on 1 m × 1 m patch repairs and is being taken to the next stage of commercial development.

## 2. Test Programme

Experimental investigations were carried out to determine the temperature development of microwave cured repair materials. Different repair materials were cast in 100 mm and 150 mm polystyrene cube moulds and a thermocouple was installed at the centroid of each cube. The internal temperature at the centroid was monitored during microwave curing and for 24 hours afterwards. The temperature at the top surface mid-point was also monitored by a thermal camera. Experimental research procedure of this study is presented in Figure 1.
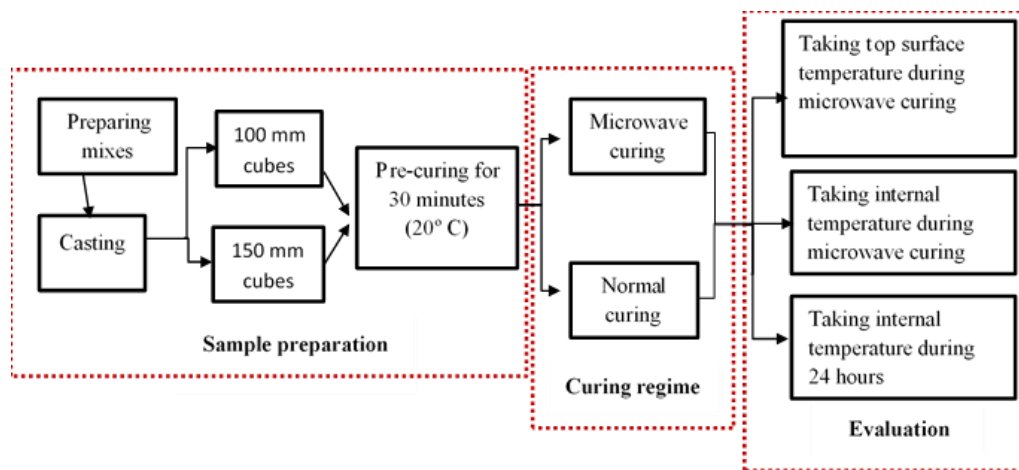


**Figure 1** Experimental procedure.

### 2.1 Repair Materials

In total six proprietary (commercial) repair materials and a CEM II mortar (Materials 1 to 7) were used for this investigation. Summary of their descriptions are presented here, however, a more detailed description can be found elsewhere [25, 26].

Material 1: Material 1 is a proprietary pre-blended shrinkage-compensated, rapid hardening cement mortar with pulverised fuel ash. Density of the fresh mix was 2200 kg/m$^3$.

Material 2: Material 2 is a proprietary polymer-modified cement mortar, fibre-reinforced and shrinkage-compensated. Density of the fresh mix was 1725 kg/m$^3$.

Material 3: Material 3 is a proprietary pre-blended polymer-modified cement based poured repair material, fibre-reinforced, shrinkage-compensated mortar. Density of fresh mix was 2250 kg/m$^3$.

Material 4: Material 4 is a proprietary lightweight, low permeability, polymer modified cement mortar. Density of the fresh mix was 1500 kg/m$^3$.

Material 5: Material 5 is a proprietary polymer-modified, low resistivity, highway agencies class M patching mortar and render for cathodic protection. Density of the fresh mix was 2200 kg/m$^3$.

Material 6: Material 6 is a proprietary polymer-modified, fibre reinforced Portland cement based fast setting repair material. Density of fresh mix was 2150 kg/m$^3$.

Material 7: Material 7 is a CEM II mortar prepared with Portland limestone cement (CEM II/A-L32.5 N) [27] and coarse sharp sand (50% passing a 600 µm sieve) in a ratio of 1:2. The mix was designed with w/c ratio of 0.5 and the plastic density of 2200 kg/m$^3$.

The repair materials were mixed according to the manufacturer's recommendations. These materials (Materials 1 to 6) are a single-component and they require only the addition of water. The properties of the repair materials are presented in Table 1. In addition, the chemical composition of the repair materials was determined by XRF (PANalytical MagiX Pro X-ray Fluorescence) and is presented in Table 2. A more detailed description of the repair materials can be found elsewhere [25, 26].

**Table 1** Properties of the repair materials.

| Repair material | W/P | 28 days compressive strength (MPa) | Adhesive bond (MPa) | Density of mix (kg/m$^3$) |
|---|---|---|---|---|
| Material 1 | 0.13 | 65 | 1.5 | 2200 |
| Material 2 | 0.14 | 48 | Class R4> 2.0 | 1725 |
| Material 3 | 0.13 | 65-70 | - | 2250 |
| Material 4 | 0.13 | 43 | 1.5 | 1500 |
| Material 5 | 0.10 | 45 | >2.0 | 2200 |
| Material 6 | 0.14 | 60 | Class R4> 2.0 | 2150 |
| Material 7 | 0.50 (w/c) | 42 | - | 2200 |

**Table 2** Chemical composition (%) of repair materials.

| Oxide | Material 1 | Material 2 | Material 3 | Material 4 | Material 5 | Material 6 | Material 7 |
|---|---|---|---|---|---|---|---|
| $Na_2O$ | 0.22 | 0.42 | - | 0.53 | 0.30 | 0.43 | - |
| MgO | 0.63 | 1.50 | 0.82 | 0.69 | 0.70 | 3.14 | 0.74 |
| $Al_2O_3$ | 7.32 | 10.70 | 10.30 | 15.50 | 3.80 | 9.67 | 3.70 |
| $SiO_2$ | 14.66 | 38.80 | 57.20 | 30.80 | 46.30 | 31.20 | 20.60 |
| $P_2O_5$ | 0.17 | - | 0.07 | - | 0.07 | 0.08 | 0.13 |
| $SO_3$ | 2.37 | 2.84 | 3.37 | 5.53 | 2.17 | 3.13 | 2.81 |
| $K_2O$ | 1.14 | 1.25 | 0.96 | 1.20 | 0.88 | 1.02 | 1.23 |
| CaO | 69.67 | 41.30 | 25.20 | 41.70 | 43.00 | 49.00 | 67.40 |
| $TiO_2$ | 0.90 | 0.23 | 0.21 | 0.32 | 0.20 | 0.20 | 0.12 |
| $Fe_2O_3$ | 2.66 | 2.73 | 1.64 | 3.55 | 2.37 | 1.66 | 3.00 |
| ZnO | 0.06 | 0.03 | 0.07 | - | - | - | - |
| SrO | 0.06 | - | 0.03 | 0.06 | 0.19 | 0.05 | 0.06 |

### 2.2 Microwave Curing Regime

Two commercial microwave ovens were used for this investigation. A Logik Model L25MDM13 oven with a maximum output power of 600 W (900 W manufacturer's specification) was used for curing the 100 mm specimens. A Sharp Model R-2370 with a maximum power of 1300 W

(manufacturer's specification also 1300 W) was used for curing the 150 mm cube specimens. Both microwave ovens could be set to generate power at incremental levels of 10% up to 100% of the maximum output power. The microwave frequency of the ovens was 2.45 GHz. A 10% power level was used for both microwaves to generate an output power of 60 W and 130 W to microwave cure 100 and 150 mm cube specimens, respectively. The microwave ovens were calibrated according to BS EN 60705 [28] and ASTM F1317 [29]. Similar approaches for microwave curing in laboratory based investigations have been adopted by other researchers [30, 31]. The fine control of power available in the microwave ovens enabled accurate development of pre-determined curing temperature [20] in a short period of time.

### 2.3 Preparation of Cube Specimens, Curing and Temperature Monitoring

Polystyrene cube moulds were used to cast specimens of the repair materials with the dual purpose of enabling curing in the microwave oven and providing insulation during the hydration period. This enabled the detection of microwave curing and hydration temperature interactions. While the polystyrene moulds do not provide idealised adiabatic conditions required for heat of hydration measurement of cements [32, 33], they are suitable to simulate an element within a repair patch of concrete. 100 mm polystyrene cube moulds (20 mm wall thickness) were used to prepare two cube specimens for each repair material. A T-type thermocouple was located at the centroid of each cube by securing it to a wooden stick as shown in Figure 2. The tip of the thermocouple was positioned at the centroid of the cube, approximately 5 mm away from the stick. Both ends of the orifices in which the T-type thermocouples were inserted were fixed to avoid water loss from the orifices. Preliminary tests were conducted to confirm no water loss. A similar method for monitoring internal temperature of concrete specimens has been used by other researchers [34].
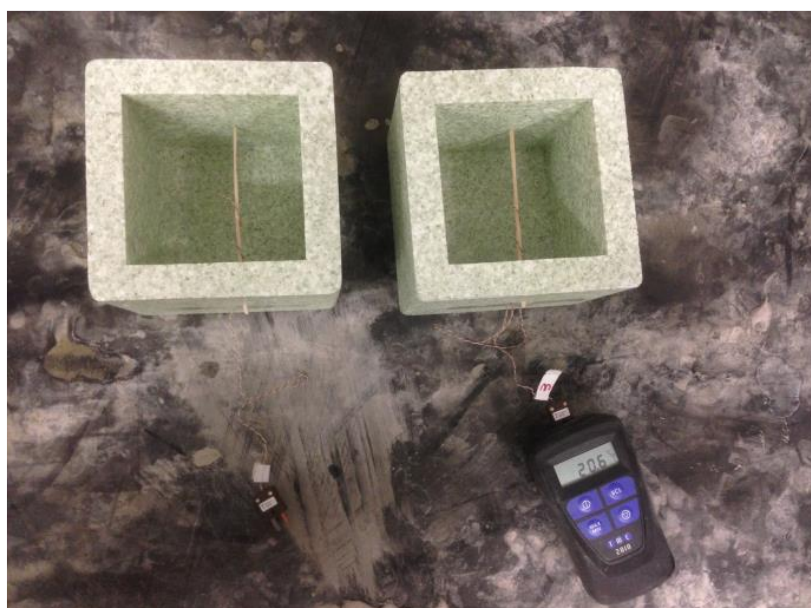


**Figure 2** Polystyrene mould with support for T-type thermocouple.

The repair materials were mixed in proportions according to the manufacturers' recommendations. The cube specimens were cast and compacted by vibration in the laboratory environment (approximately 20 °C and 60% RH). After 30 minutes from the start of mixing, one of

the two cube specimens were selected as control and cured in the ambient environment (20 °C, 60% RH) up to 24 hours from the start of mixing. The second cube specimen of each repair material was selected for microwave curing at 60 Watts starting 30 minutes from commencing mixing to reach a target temperature of 40-45 °C.

Flir i7 thermal camera and Data Taker DT85G were used to record the top surface and internal temperature of the cube specimens respectively. These temperatures were recorded before the start of microwave curing and then at every 10 minutes interval until the end of microwave curing. Similar methods of monitoring internal and surface temperatures of concrete specimens have been used by other researchers [9]. The microwave cured cubes were stored in the laboratory environment along with the normally cured control specimens. The internal temperatures of both cubes were continuously recorded up to 24 hours using Data Taker DT85D.

A previous study [20] reported that a different time period of microwave curing is required for different repair materials to achieve a similar target temperature since each repair material has a different capacity to absorb microwave energy [20]. The absorption of microwave energy in general is related to the dielectric properties of each material, temperature of the material, intensity of the electric field inside the microwave cavity and the constituents of repair materials such as admixtures, additives and the fineness of powder as well as w/c ratio [35]. However, for the current study, in order to reach target temperature, the cube specimens were microwave cured between 40 to 50 minutes with the exception of Material 6 which was microwave cured only for 15 minutes. Table 3 shows the microwave curing period and the top surface temperature of all repair materials exposed to 60 Watts.

In addition, Materials 1 and 7 mixes were also cast in 150 mm polystyrene cube moulds and exposed to microwave curing at a power of 130 W for 45 minutes. Similar to the 100 mm cubes, the temperature at the centroid and top surface was measured during microwave curing by a thermocouple and a thermal camera, respectively. The internal temperature at the centroid was recorded for 24 hours after casting. These specimens were used to investigate the effect of specimen size on the heat of hydration.

**Table 3** Microwave curing period for cubes of different repair materials.

| Repair Materials | Cube size (mm) | Microwave Power (Watts) | Microwave curing period (mins) | Top surface temperature (°C) |
|---|---|---|---|---|
| Material 1 | 100 | 60 | 50 | 39.9 |
| Material 2 | 100 | 60 | 40 | 40.7 |
| Material 3 | 100 | 60 | 40 | 41.8 |
| Material 4 | 100 | 60 | 40 | 44.1 |
| Material 5 | 100 | 60 | 45 | 41.5 |
| Material 6 | 100 | 60 | 15 | 40.7 |
| Material 7 | 100 | 60 | 50 | 42.1 |
| Material 1 | 150 | 130 | 45 | 43.1 |
| Material 7 | 150 | 130 | 45 | 43.2 |

## 3. Results and Discussion

### *3.1 Temperature Increase in Microwave Cured Specimens*

3.1.1 Surface and Internal Temperature during Microwave Curing

Figure 3 shows the internal temperature at the centroid and top surface for microwave cured Material 7. The 150 mm cube specimen was exposed to 130 W of microwave energy for 45 minutes to reach microwave curing temperature of 43.2 °C at the top surface. The internal temperature recorded by thermocouples at the centroid of the specimen before the start of microwave heating (at 0 minutes, Figure 3) is 21 °C. The top surface temperature recorded by the thermal camera is lower (17 °C), similar to the room temperature. The internal and surface temperatures increase linearly with curing time, remaining within 10% of each other. Similar temperature profiles were also observed for the 150 mm cubes of Material 1 during microwave curing. These results indicate that monitoring the top surface temperature by the thermal camera provides a reasonably accurate representation of the internal temperature at the centroid. Practical repairs normally have a larger volume and surface area than the 150 mm cube specimens. Field tests on scaled up repairs also show that the top surface temperature, monitored by thermal camera, provides reliable representation of the internal temperature under microwave curing [15].

Analytical simulations done by researchers [18, 19, 36-38] at different depths, mainly in food products, showed a decrease in microwave power with depth and hence a higher top surface temperature. Most of these analytical studies assumed a maximum thickness of 20-30 mm which is much less than the 150 mm cubes discussed here. The simulations exposed the solids on one or two sides [39] to high power for a few minutes, whereas the concrete cubes were exposed to microwaves on all faces at low power for 40-50 minutes. The centroid of the cubes represented the maximum distance (75 mm) from the faces of the cubes which were exposed to microwaves.
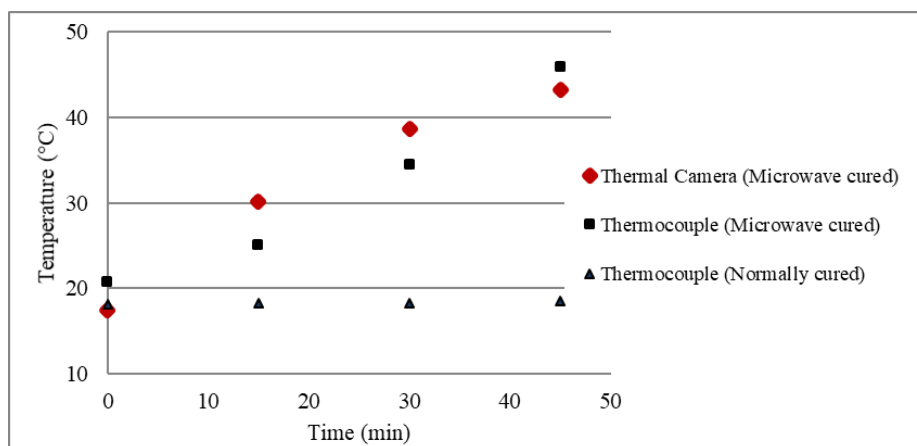


**Figure 3** Temperature development during microwave curing for Material 7 (150 mm cube).

3.1.2 Internal Temperature Development during 24 Hours after Microwave Curing

Figures 4(a-g) show the internal temperature profiles (at the centroid of 100 mm cubes) for normally and microwave cured specimens of all repair materials during 24 hours after commencing

mixing. Additional small graphs have also been added to the main temperature profiles in order to show in detail the internal temperature profile over a short period at the end of microwave curing for materials 2, 4 and 6 (Figures 4b,d,f). The initial linear part of the graphs for microwave curing (MC) represents the MC period and shows the peak MC temperature for each material (Table 3). All graphs show that the normally and microwave cured specimens achieved their peak of hydration temperature after the MC period at different times during the 24 hours due to differences in their constituents [40, 41].
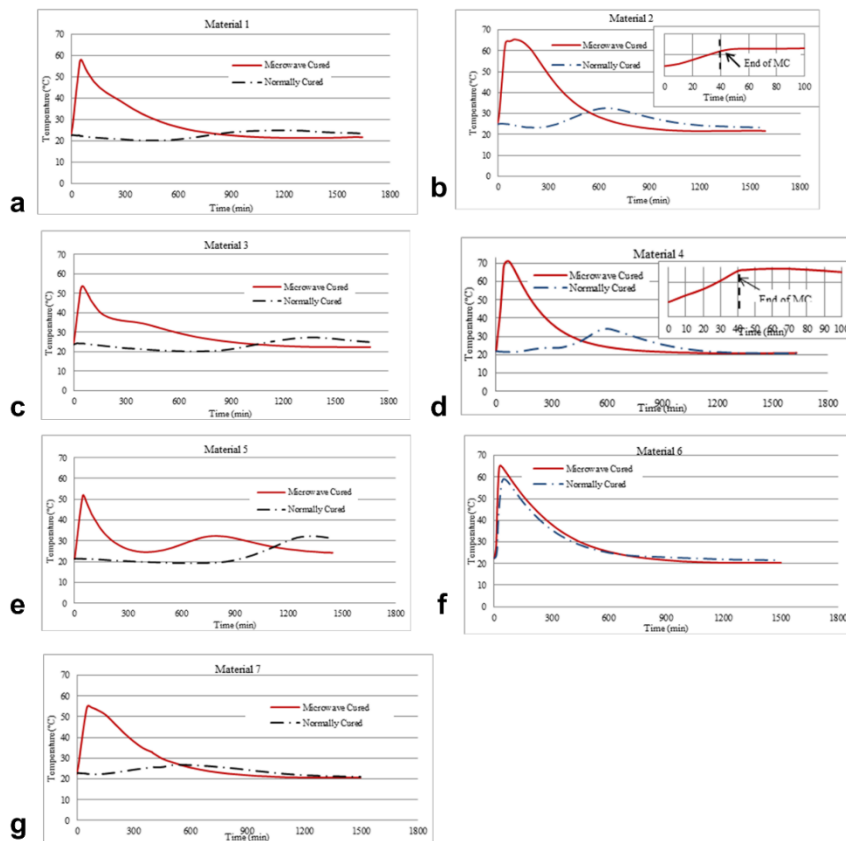


**Figure 4** Internal temperature of microwave and normally cured Materials.

Microwave curing impacts the heat of hydration developed after attaining the maximum microwave curing temperature, as shown in Figures 4, by reaching the subsequent peak temperature much earlier than the corresponding normally cured specimen for each repair material. This is due to the acceleration of hydration caused by high temperature [42]. The internal temperature of all materials continued to rise gently for up to 10 minutes immediately after the end of microwave curing due to temperature redistribution from the hotter zones, except the fast setting material 6 (Figure 4f) where the temperature rise was much sharper due to the acceleration of early hydration temperature caused by microwave heating. The peak heat of hydration almost merges or is close to the end of microwave curing for some repair materials such as materials 4 and 6 (Figure 4d and Figure 4f) for which the peak heat of hydration temperature occurred at 15-20 minutes after the end of microwave curing and remained constant for about 15 minutes. The normally cured specimen of repair Material 6 (Figure 4f) had a very similar heat of hydration temperature profile as the microwave cured specimen due to its high MgO content (Table 2) which

provides rapid hardening properties [43]. For instance, the hydration temperature of the microwave cured Material 6 of normal density (2150 kg/m$^3$) peaked at about 15 minutes after the end of microwave curing (60 minutes after start of mixing) whereas the hydration temperature of the corresponding normally cured Material 6 peaked at 80 minutes (Figure 4f) after the start of mixing. Material 6 is based on rapid setting, rapid hardening Portland cement which emits more heat during the initial setting time of about 30 minutes. The accelerated heat of hydration under microwave curing of material 4 may be caused by its relatively high SO$_3$ and Al$_2$O$_3$ content [44].

A comparison of the control specimens of materials 1 and 6, which are both based on rapid setting, rapid hardening Portland cement, shows that material 1 has a more normal heat of hydration profile due to the presence of fly ash whose pozzolanic reactions delay hydration. The temperature profile of the control specimen of Material 6 (fast setting repair material) is very similar to the results reported by Barde et al. [45] for a rapid setting cement based material. However, the temperature profiles of the microwave cured samples of both materials 1 and 6 are similar since the pozzolanic reactions of fly ash are accelerated by microwave curing.

The peak heat of hydration also almost merged with the end of microwave curing in materials 2 (Figure 4b) and 4 (Figure 4d) at about 60 and 20 minutes respectively after the end of microwave curing. The hydration temperature of the corresponding normally cured materials 2 and 4 peaked at about 610 and 560 minutes after the end of microwave curing. Both 2 and 4 are Portland cement based polymer modified lightweight materials (density 1725 and 1500 kg/m$^3$), whose heat of hydration is accelerated by microwave curing.

The normal density Portland cement based repair materials 3, 5 and 7, with and without polymer modification, reached their peak heat of hydration during the post microwave curing period. This occurred during the cooling down period after microwave curing of the 100 mm cube specimens, although for material 7 in Figure 4g the peak position is not distinct due to the small size of test specimens. This becomes clear in Figure 5 where the corresponding data of the 150 mm cube specimens show that the temperature continued to rise due to heat of hydration after the end of microwave curing. It reached a maximum of 73.1 °C at 165 minutes after the start of microwave curing. The microwave cured Material 5 shows a peak temperature (Figure 4e) of 32.5 °C at approximately 800 minutes after the start of mixing. The normally cured sample of Material 5 also reached the maximum heat of hydration temperature of 32.3 °C at approximately 1350 minutes after the start of mixing. The longer dormant periods of both normally cured materials 3 and 5 are due to admixtures used in them whereas material 7 is admixture-free and has a very short dormant period [46]. The length of these dormant periods has a significant effect on the hydration temperature profiles under microwave curing as discussed in section 3.2.2.
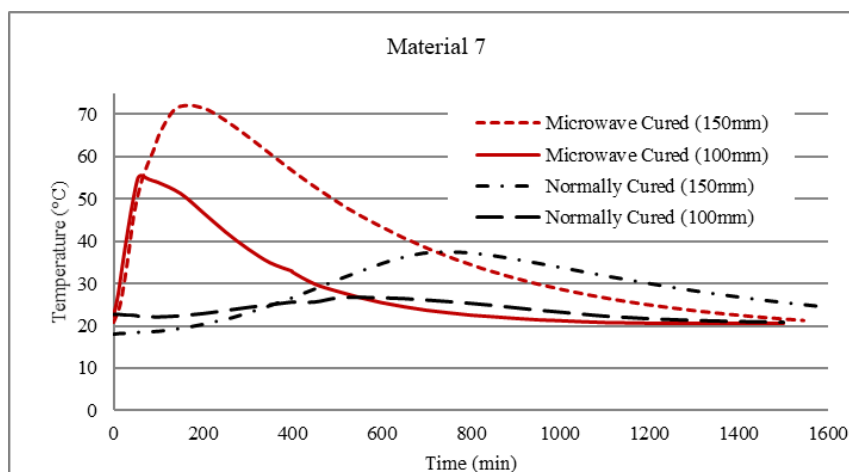
**Figure 5** Internal temperature of microwave and normally cured Material 7 for 100 and 150 mm cubes.

Figures 4b, 4d and 4g show that the normally cured repair materials 2, 4 and 7 have a short dormant period (200 to 300 minutes) before the hydration temperature starts rising. This relates to the peak hydration temperature of the corresponding microwave cured specimens occurring soon after the end of microwave curing. On the other hand, normally cured materials 1, 3 and 5 in Figures 4a, 4c and 4e show a long dormant period (600 to 900 minutes) before hydration temperature rises. In these cases, the peak hydration temperature of the corresponding microwave cured specimens takes longer. It occurs during the cooling down period after microwave curing in the 100 mm cube specimens but in larger volume repairs (e.g. 150 mm cubes) in Figures 5 and 6 it continues to rise until the peak hydration temperature is reached and then starts falling. The longer dormant periods in Figures 4a, 4c and 4e reflect the composition of the normal density repair materials 1, 3 and 5 where cement replacement materials and admixtures have prolonged the dormant period. Material 1 contains pfa while Materials 3 and 5 are polymer modified, both additives delay the setting time and early hydration [46].

The results presented here are in agreement with other researchers who investigated the effect of temperature on heat of hydration [2, 47] of different cements cured at elevated temperature with conventional heat. For example, Hill et al. [47] reported the peak heat evolved and the time it occurs for OPC hydrated at various temperatures (20, 40 and 90 °C) by means of isothermal conduction calorimetry. They reported that increasing curing temperature results in a higher heat of hydration peak which occurs earlier. For example, OPC hydrated at 20 °C shows a peak of heat above 3.5 W/kg after about 12 hours. The corresponding peak of heat is 11 W/kg after about 5 hours for OPC cured at 40 °C. However, under conventional thermal curing used by the researchers [2, 47], heat was applied throughout the experiment (approximately 24 hours) to keep the elevated curing temperature constant whereas microwave heating in this investigation was applied only for a short time (up to 50 minutes). For example, normally cured Material 7 (CEM II mortar), shows a maximum internal temperature of 26.7 °C at 630 minutes (10.5 h) after casting. The corresponding temperature for microwave cured CEM II mortar is 55.4 °C at 90 minutes (1.5 h) after casting. This is a significant result which indicates that even a short period of microwave heating at early age can have a similar effect as a much longer period of continuous heating [44] on the peak heat of hydration and the time it occurs.

The temperature profiles in Figure 4 show that all repair materials attain a significantly higher maximum temperature than the microwave curing temperatures, given in Table 3 (39.9 - 44.1 °C), reached at the end of the linear part of the graph. These values range between 51 to 70 °C. It is clear that the heat of hydration will have a significant effect on the temperature of early age repairs which in practice will have a much larger volume and surface area than the small cube specimens. The heat of hydration increases with increasing volume of concrete [48] while the higher surface area of repairs will help in controlling it. This is likely to impact long term durability due to delayed ettringite formation and early shrinkage cracking. Therefore, the permissible microwave curing temperature should consider this cumulative effect of the hydration temperature by adopting the following equation [20] to prevent curing temperature exceeding 70 °C:

$$T_m + \Delta T_h \leq \frac{70}{\gamma_T} \tag{1}$$

$$\Delta T_h = T_h - T_m \tag{2}$$

where, as shown in Figure 6, $T_m$ is the permissible temperature at the end of microwave curing (°C); $T_h$ is the peak heat of hydration temperature of unhardened concrete (°C); $\gamma_T$ is the factor of safety accounting for microwave curing temperature variations (hot spots).
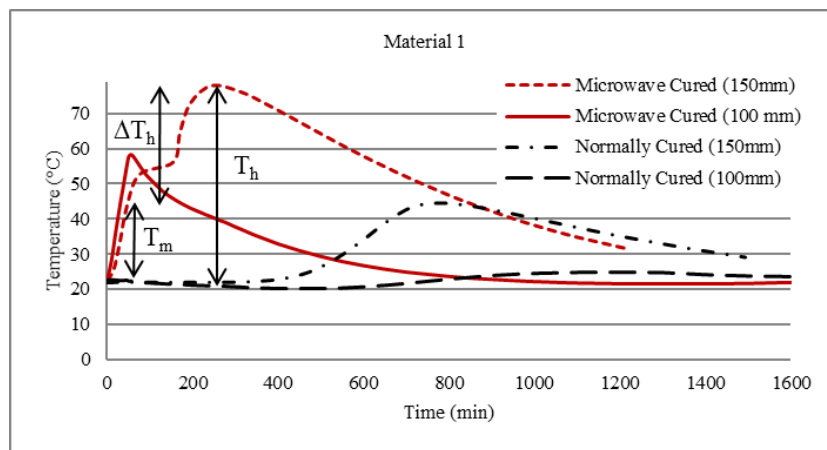


**Figure 6** Internal temperature of microwave and normally cured Material 1 for 100 and 150 mm cubes.

### 3.2 Influence of Volume on the Heat of Hydration

3.2.1 Normally Cured Specimens

A comparison between the 100 mm and 150 mm normally cured cube specimens of volume 1 and 3.4 litres, respectively, shows that increasing the size of the specimen results in more heat of hydration. For example, the 100 mm cube of material 7 achieves the peak heat of hydration temperature of 26.7 °C (Figure 5). The corresponding result for the 150 mm cube specimen was 37.8 °C (Figure 5). The trend is similar for Material 1 with the 100 mm cube showing a peak temperature of 24.9 °C (Figure 6) compared with 45.1 °C (Figure 6) for the 150 mm cube. This is in agreement with the results reported by Lee et al. [48] from adiabatic tests on OPC concrete cylinders of 6, 30 and 50 litre volumes. Their results showed that the higher volume results in higher adiabatic

temperature rise, achieving a temperature rise of below 50 °C for a 6 litre volume and 60 °C for a 30 litre volume.

The effect of volume on the heat of hydration is well documented and it is an important issue when considering a mass concrete application. However, in the case of patch repairs the volume/surface ratios are small since repair patches have a low thickness and relatively large surface area. Consequently, heat of hydration is unlikely to be a problem under normal curing but needs to be addressed under microwave curing since the maximum temperature could exceed the durability limit of about 70 °C [20].

### 3.2.2 Microwave Cured Specimens

The temperature rise after microwave curing of the 100 mm and 150 mm cubes of Materials 1 and 7 is shown in Figures 5 and 6. The internal temperature at the centroid of the cubes is recorded in these figures. Figure 5 shows that at about 10 minutes after the end of microwave curing the temperature of the 100 mm cube of Material 7 starts falling steadily without showing any significant uplift due to the heat of hydration. This is due to the small size (volume) of the cube which does not allow sufficient build-up of the heat of hydration. However, Figure 5 shows that the corresponding temperature for the 150 mm cube continued to rise after the end of microwave curing to reach a maximum value of 73.1 °C after 165 minutes from the start of microwave curing. This is more representative of practical repair patches which have a relatively large volume. The corresponding results for the Material 1 in Figure 6 show a similar trend with the peak heat of hydration temperature of 58.2 and 78.2 °C occurring at 55 and 245 minutes for the 100 and 150 mm cubes respectively. A comparison between the time of peak hydration temperature reached by the normally and microwave cured specimens shows the acceleration of hydration by microwave curing. The peak times are 550 (normally cured) and 60 (microwave cured) minutes respectively for 100 mm cubes compared with 754 (normally cured) and 165 (microwave cured) minutes for 150 mm cubes.

The hydration profile of the normally cured material 1 in Figures 6 shows a long dormant period followed by a sharp rise in the heat of hydration of the 150mm cube. The dip in the rate of temperature increase for a short period immediately after the end of microwave curing for the 150mm cube of material 1 in Figures 6 is due to the long dormant period of the normally cured 150mm cube. This dip is shortly followed by a sharp increase in the rate of temperature rise in the microwave cured cube which reflects the sharp rise in the heat of hydration after the long dormant period of the corresponding normally cured 150 mm cube of material 1. The dormant period for the normally cured material 7 (Figure 5) is significantly shorter and results in providing a steady rate of temperature rise (without a dip) beyond the end of microwave curing for the corresponding 150mm cube of material 7.

Considering the 150 mm cube data in Figure 6, the difference between the peak hydration temperature $T_h$ and the temperature at the end of microwave curing $T_m$ gives a measure of the temperature provided by heat of hydration under microwave curing, $\Delta T$. This is compared with the heat of hydration temperature of the normally cured samples which is given by the difference between the peak hydration temperature $T_h$ and $T_{datum}$ (laboratory temperature of 17.6 °C). In the case of material 1, the value of $T_h - T_m = 34$ °C whereas for the normally cured sample $T_h - T_{datum} = 22.5$ °C. Similarly, for material 7, $T_h - T_m = 26.2$ °C whereas $T_h - T_{datum} = 19.5$ °C. These results show

the significant increase caused by microwave heating in the hydration temperature of the repair materials. Similarly, Escalante-Garcia and Sharp [2] showed that higher curing temperature of OPC accelerates hydration and results in higher heat evolution.

The heat of hydration contributes significantly to the total temperature developed at early age by the microwave cured repair materials. Its implications on the long term durability can be serious. Many researchers have focussed on microwave curing to develop high strength of concrete at early age. Microwave curing temperatures in the range 60 to 80 °C have been used by the researchers [8, 9, 49] without considering the cumulative effect of heat of hydration on the peak temperature and its impact on durability. This impact is likely to be more significant when microwave curing is used under normal ambient temperatures than under cold weather conditions. Recent research has shown much greater benefit of microwave curing in cold weather conditions [21].

## 4. Conclusions

The conclusions pertain to proprietary concrete repair materials microwave cured to a surface temperature of about 40 °C for 40-45 minutes (except the fast setting material 6 which was cured for 15 minutes). The tests reported in this paper represent semi-adiabatic conditions of hydration of small volumes (100 and 150 mm cubes) of repair mortars cast in insulated polystyrene moulds.

A short period of early age microwave curing results in an increase in the hydration temperature and brings forward the peak heat of hydration time relative to the control specimens which are continuously exposed to ambient conditions (20 °C, 60% RH).

The peak heat of hydration of normal density (about 2200 kg/m$^3$) rapid hardening Portland cement based repair materials with either pfa or polymer addition almost merges with the end of microwave curing period. Similarly, lightweight (density 1500-1700 kg/m$^3$) cement based polymer modified repair materials also develop heat of hydration rapidly which almost merges with the end of microwave curing period.

The peak heat of hydration of normal density ordinary Portland cement based repair materials, with and without polymer addition, occurs during the post microwave curing period.

The short period of early age microwave curing also increases the peak hydration temperature. The sum of the microwave curing temperature and the subsequent heat of hydration temperature can easily exceed the limit of about 70 °C early age curing temperature beyond which durability problems such as delayed ettringite formation and cracking can become inherent. Limits to the maximum microwave curing temperature are required to prevent this problem, which will depend on the cementitious binder composition. The maximum microwave curing temperature was limited to about 40 °C for this study.

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**Competing Interests**

The authors declare that competing interests do not exist.

**References**

1. Kumar M, Singh SK, Singh NP. Heat evolution during the hydration of Portland cement in the presence of fly ash, calcium hydroxide and super plasticizer. Thermochim Acta. 2012; 548: 27-32.
2. Escalante-Garcia JI, Sharp JH. The effect of temperature on the early hydration of Portland cement and blended cements. Adv Cem Res. 2000; 12: 121-130.
3. Copeland LE, Kantro DL. Hydration of Portland cement. Proceedings of the 5th International Symposium on the Chemistry of Cement, Tokyo, Japan, 1968; 2: 387-421.
4. Kjellsen KO, Detwiler RJ. Reaction kinetics of Portland cement mortars hydrated at different temperatures. Cem Concr Res. 1992; 22: 112-120.
5. Zhang JY, Cusson D, Monteiro P, Harvey J. New perspectives on maturity method and approach for high performance concrete applications. Cem Concr Res. 2008; 38: 1438-1446.
6. Pichler C, Schmid M, Traxl R, Lackner R. Influence of curing temperature dependent microstructure on early-age concrete strength development. Cem Concr Res. 2017; 102: 48-59.
7. Gallucci E, Zhang X, Scrivener KL. Effect of temperature on the microstructure of calcium silicatehydrate (C-S-H). Cem Concr Res. 2013; 53: 185-195.
8. Leung CK, Pheeraphan T. Determination of optimal process for microwave curing of concrete. Cem Concr Res. 1997; 27: 463-472.
9. Kong Y, Wang P, Liu S, Gao Z. Hydration and microstructure of cement-based materials under microwave curing. Constr Build Mater. 2016; 114: 831-838.
10. Makul N, Agrawal DK. Microwave-accelerated curing of cement-based materials: Compressive strength and maturity modeling. Key Eng Mater. 2011; 484: 210-221.
11. Kong Y, Liu S, Wang P. Effects of microwave curing on the compressive strength development and hydration of cement-granulated blast furnace slag composite system. Constr Build Mater. 2021; 270. doi: 10.1016/j.conbuildmat.2020.121432.
12. Choi H, Koh T, Choi H, Hama Y. Performance evaluation of precast concrete using microwave heating form. Materials. 2019; 12: 1113.
13. Łaźniewska-Piekarczyk B. The influence of chemical admixtures on cement hydration and mixture properties of very high performance self-compacting concrete. Constr Build Mater. 2013; 19: 643-662.

14. Makul N, Rattanadecho P, Agrawal DK. Applications of microwave energy in cement and concrete – a review. Renew Sust Energ Rev. 2014; 37: 715-733.

15. Mangat PS, Grigoriadis K, Abubakri S, Javaid A, Zhao C. Microwave system for in-situ curing of concrete repair. in: Concrete solutions: Proceedings of Concrete Solutions 6th International Conference on Concrete Repair, Thessaloniki, Greece, 20-23 June 2016. Boca Raton, Florida: CRC Press; 2016.

16. Makul N, Rattanadecho P, Pichaicherd A. Accelerated microwave curing of concrete: A design and performance-related experiments. Cem Concr Compos. 2017; 83: 415-426.

17. Bai Y, Shi S, Fabian M, Sun T, Grattan K, McKinnon B, McCague C. Potential of microwave curing for precast concrete manufacture. Institute of Concrete Technology Yearbook. 2016: 61-66. Available from: https://openaccess.city.ac.uk/id/eprint/17170/.

18. Wei W, Shao ZS, Chen WW, Zhang PJ, Yuan Y. A fully coupled electromagnetic irradiation, heat and mass transfer model of microwave heating on concrete. IEEE Access. 2021; 9: 1575-1589.

19. Rattanadecho P, Suwannapum N, Cha-um W. Interactions between electromagnetic and thermal fields in microwave heating of hardened type i-cement paste using a rectangular waveguide (influence of frequency and sample size). J Heat Transfer. 2009; 131: 1-12.

20. Mangat PS, Grigoriadis K, Abubakri S. Microwave curing parameters of in-situ concrete repairs. Constr Build Mater. 2016; 112: 856-866.

21. Grigoriadis K, Mangat PS, Abubakri S. Bond between microwave cured repair and concrete substrate. Mater Struct. 2017; 50: 1-14.

22. Mangat PS, Abubakri S, Grigoriadis K. Bond of steel reinforcement with microwave cured concrete repair mortars. Mater Struct. 2017; 50: 1-16.

23. Zhao W, Su Q, Han F, Wang W. Study on the heat of hydration and strength development of cast-in-situ foamed concrete. Adv Mater Sci Eng. 2020; 2020: 1-12.

24. Taylor HF, Famy C, Scrivener KL. Delayed ettringite formation. Cem Concr Res. 2001; 31: 683-693.

25. Mangat PS, Grigoriadis K, Abubakri S. Temperature development in microwave cured repair materials. Proceedings of the Concrete Solutions, 6th International Conference on Concrete Repair; 2016 June 20-23; Thessaloniki, Greece. Boca Raton, Florida: CRC Press.

26. Mangat PS, Grigoriadis K, Abubakri S. Microwave curing of concrete bridge repairs. Proceedings of the 16th European Bridge Conference; 2015 June 23-25; Edinburgh, UK.

27. BS EN 197-1. Cement- Part 1: Composition, specifications and conformity criteria for common cements [Internet]. London, UK: British Standards Institution; 2011. Available from: https://global.ihs.com/doc_detail.cfm?document_name=BS%20EN%20197%2D1&item_s_key=00358162.

28. BS EN 60705. Household microwave ovens. Methods for measuring performance [Internet]. London, UK: British Standards Institution; 2012. Available from: https://webstore.ansi.org/standards/bsi/bsen607052012.

29. ASTM F1317-98. Standard test method for calibration of microwave ovens [Internet]. West Conshohocken, PA: ASTM International; 2012. Available from: https://standards.globalspec.com/std/3848136/astm-f1317-98-2012.

30. Wang Y, Luo S, Yang L, Ding Y. Microwave curing cement-fly ash blended paste. Constr Build Mater. 2021; 282: 1-9.

31. Wu D, Sun W, Liu S, Qu C. Effect of microwave heating on thermo-mechanical behavior of cemented tailings backfill. Constr Build Mater. 2021; 2: 121180.

32. BS EN 196-9. Methods of testing cement. Heat of hydration. Semi-adiabatic method [Internet]. London, UK: British Standards Institution; 2010. Available from: [https://www.en-standard.eu/bs-en-196-9-2010-methods-of-testing-cement-heat-of-hydration-semi-adiabatic-method/](https://www.en-standard.eu/bs-en-196-9-2010-methods-of-testing-cement-heat-of-hydration-semi-adiabatic-method/).

33. TCE1: Adiabatic and semi-adiabatic calorimetry to determine the temperature increase in concrete due to hydration heat of the cement. Mater Struct. 1997; 30: 451-464.

34. Pan Y, Zhang Y, Li S. Effects of isothermal microwave curing on steel fibre-reinforced reactive powder concrete: Strength, microstructure and hydration products. Constr Build Mater. 2021; 302: 124435.

35. Kharkovsky SN, Hasar UC, Atis CD, Döver S. Electromagnetic properties of cement-based materials over time at microwave frequencies [Internet]. Available from: [https://www.emo.org.tr/ekler/b922393a3cb4627_ek.pdf](https://www.emo.org.tr/ekler/b922393a3cb4627_ek.pdf).

36. Yang HW, Gunaskearan S. Temperature profiles in a cylindrical model food during pulsed microwave heating. J Food Sci. 2006; 66: 998-1004.

37. Campañone LA, Paola CA, Mascheroni RH. Modeling and simulation of microwave heating of foods under different process schedules. Food Bioproc Tech. 2012; 5: 738-749.

38. Brodie G. The influence of load geometry on temperature distribution during microwave heating. Trans ASABE. 2008; 51: 1401-1413.

39. housova J, Hoke K. Temperature profiles in microwave heated solid foods of slab geometry: Influence of process parameters. Czech J Food Sci. 2001; 19: 111-120.

40. Cheeseman CR, Asavapisit S. Effect of calcium chloride on the hydration and leaching of lead-retarded cement. Cem Concr Res. 1999; 29: 885-892.

41. Cheung J, Jeknavorian A, Roberts L, Silva D. Impact of admixtures on the hydration kinetics of Portland cement. Cem Concr Res. 2011; 41: 1289-1309.

42. Neville AM. Properties of concrete. 5th ed. Harlow, Essex: Pearson Education Limited; 2012.

43. Seehra Ms SS, Gupta S, Kumar S. Rapid setting magnesium phosphate cement for quick repair of concrete pavements — characterisation and durability aspects. Cem Concr Res. 1993; 23: 254-266.

44. Yamashita M, Harada T, Saka E, Tsuchiya K. Influence of sulfur trioxide in clinker on the hydration heat and physical properties of Portland cement. Constr Build Mater. 2020; 250; 1-10.

45. Barde A, Parameswaran S, Chariton T, Weiss W, Cohen MD, Newbolds S. Evaluation of rapid setting cement-based materials for patching and repair. Purdue University; West Lafayette, IN; 2006; FHWA/IN/JTRP/2006-11.

46. Kang S, Kim J, Moon C, Song M. Early hydration-retarding mechanism of polymer-modified cement. Mater Res Innov. 2015; 19: S8-22-S8-26.

47. Hill J, Whittle BR, Sharp JH, Hayes M. Effect of elevated curing temperature on early hydration and microstructure of composite cements. MRS Online Proceedings Library. 2002; 757. doi: 10.1557/PROC-757-II10.3.

48. Lee BJ, Bang JW, Shin KJ, Kim YY. The effect of specimen size on the results of concrete adiabatic temperature rise test with commercially available equipment. Materials (Basel). 2014; 7: 7861-7874.

49. Al Bakri Abdullah MM, Mohd Tahir MF, Hussin K, Mohammed B, Ekaputri JJ. Effect of microwave curing to the compressive strength of fly ash based geopolymer mortar. Mater Sci Forum. 2016; 841: 193-199.

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