



# Production of high-early-compressive-strength Portland cement paste using low-pressure microwave-accelerated heating and curing: processing characteristics and factors affected



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## ABSTRACT

The use of low-pressure microwave (MW)-accelerated heating and curing in the production of high-early-strength Portland cement paste (CP) in relation to the processing characteristics and factors affected is investigated. The effects of the pressure in the MW cavity, the feed direction of the MW, and the number of CP specimens per MW curing batch on the temperature increase and moisture content (i.e., the water-cement ratio (w/c) of the CP and its compressive strength after MW-curing CP) are considered. A double-feed MW-vacuum system is used to generate MW by applying 800 W (1 magnetron) or 1,600 W (2 magnetrons) and to convey MW to the CP specimens. The CP pastes are designed and mixed at specific initial w/c ratios of 0.25, 0.35, and 0.45. The CP specimens are cured using a low-pressure MW cavity at 30 and 50 kPa and fed in symmetrically or asymmetrically perpendicular directions of the CP specimens in batches of 12 (3.95% of the volume of microwave cavity) and 24 specimens (7.90% of the volume of the microwave cavity) per MW-curing batch with the following dimensions: 5 cm long × 5 cm wide × 10 cm thick. The experimental results show that with the maximum MW curing temperature of 70 °C from the initial stage (approximately the first 30 min of applying low-pressure MW curing) until 100 min, the temperature of the CP increases continuously at a high rate; then, the rate at which the temperature increases decreases slightly, which is consistent with the remaining w/c of the CP. The pressure in the MW has a slightly different temperature increase and remaining CP. A perpendicular symmetric magnetron placement in respect to the horizontal position of the specimens can lead to a steady increase in the strength development of all the CP specimens. Further, over the course of 28 days, compared with the water-cured CP, the MW-cured CP develops more compressive strength.

## 1. Introduction

More than 40 years ago, microwave energy (MW) in concrete composites was proven as a potential technique to improve the properties of concrete. The applications of MW in concrete materials can be classified into three principal aspects as follows [1, 2, 3, 4]:

- **Group I:** use of MW to study the properties of concrete, such as characterizing Portland concrete [5], predicting the compressive strength of normal concrete [6] assessing the w/c of rapid setting concrete [7], reflecting the near-field measurement to

nondestructively test the concrete [8], and monitoring the water infiltration level of the concrete roof [9].

- **Group II:** use of MW for accelerated curing to improve the mechanical properties of concrete, such as the accelerated curing of mortar [10], curing of the fresh concrete incorporated using the MW-vacuum technique to enhance the compressive strength of concrete [11, 12] in the low-pressure state [13, 14] and with concrete mixed with slag powder.
- **Group III:** use of MW to destroy or recover the concrete and supplementary concrete materials such as MW treatment metakaolin [15], decommission the concrete-contaminated reinforced concrete

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**Table 1**  
Chemical composition and physical properties of OPC.

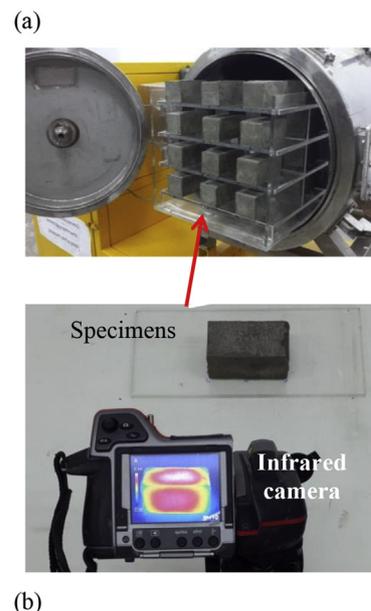
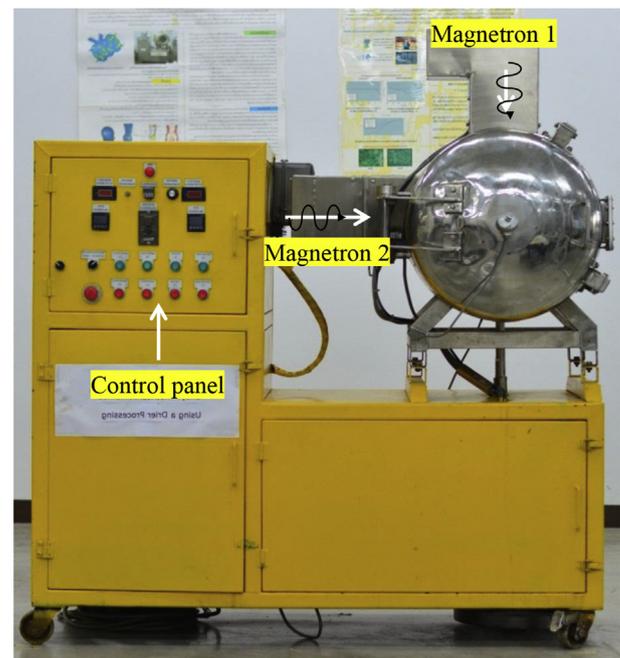
Chemical composition	OPC (Mean $\pm$ Standard deviation) (% by mass)
SiO <sub>2</sub>	19.84 $\pm$ 0.52
Al <sub>2</sub> O <sub>3</sub>	4.21 $\pm$ 0.28
Fe <sub>2</sub> O <sub>3</sub>	3.19 $\pm$ 0.09
MgO	1.25 $\pm$ 0.02
CaO	68.02 $\pm$ 1.06
Na <sub>2</sub> O	0.18 $\pm$ 0.03
K <sub>2</sub> O	0.33 $\pm$ 0.06
SO <sub>3</sub>	1.59 $\pm$ 0.17
Loss on ignition	0.92 $\pm$ 0.04
Physical properties	OPC (Mean $\pm$ Standard deviation)
Particle size at 50% cumulative passing ( $\mu$ m)	13.50 $\pm$ 0.39
Specific gravity	3.15 $\pm$ 0.02
Specific surface area (m <sup>2</sup> /kg)	825 $\pm$ 14.02

slab [16], improve the high-reactivity rice husk ash for use as a partial cement substitution material [17], etc.

The popularity of MW on concrete relies on the MW heating mechanism. When dielectric concrete materials are subjected to MW radiation, MW can penetrate into the internal structure of concrete and induce the polarization and depolarization switching of molecules of concrete constituents (especially water molecules), which causes a friction force near the surrounding molecules and generates heat from inside the materials [1]. This MW heat generation is more efficient than the conventional heating mechanism, which relies on the heating transfer mechanism in the conduction mode and depends on the thermal conductivity of concrete, which is low [18]. The low thermal conductivity affects the heat transfer proficiency, makes the heat distribution in concrete uneven (such as hot spots), and causes cracking, which affects the long-term properties and durability of the concrete.

Hydraulic cement (or Portland cement) concrete consists of water to react with cement particles and form the calcium silicate hydrate (C–S–H) structure, which improves the mechanical properties. Water is highly responsive to curing by MW. When fresh concrete is subjected to MW, the water molecules in concrete are heated and converted to vapor (moisture with high pressure), which consequently moves outward. This phenomenon causes the pores to collapse and the internal concrete structure to become denser, which enhances the compressive strength development of the concrete [19, 20].

Accelerated MW curing is a kind of accelerated curing that improves concrete depending on three conditions: concrete properties (particularly the w/c ratio), the delay time (time after mixing until the MW curing begins), and the MW generation and transfer system [2, 21]. Therefore, this study focuses on accelerated curing of CP using low-pressure MW to produce CP. The scope of the investigation includes the initial w/c (0.25, 0.35, and 0.45) of CP, which is determined based on the w/c required for a full hydration reaction of 0.25-w/c (lower margin) in accordance with the stoichiometric water consumption of the full hydration reaction [12]. A w/c of 0.45 (upper margin) is needed for a full hydration reaction with combined water and gel water required for the preceding hydration reaction and compressive strength development [12]. A w/c of 0.35 is determined to be the middle point between these margins. Also taken into account are the following: the pressure level in the MW cavity (30 and 50 kPa compared to the normal air pressure (101.325 kPa)), the feed direction of the MW energy (the placement of the CP specimen as in either a symmetrically or asymmetrically perpendicular direction in relation to microwave radiation, and the number of CP specimens per batch of MW curing (12 specimens in one batch and 24 specimens in another, all with dimensions of 5 cm long  $\times$  5 cm wide  $\times$  10 cm thick) in respect to the temperature increase and the moisture content of the remaining w/c). The laboratory-scaled double-feed MW-vacuum system



**Fig. 1.** (a) Double-feed MW and vacuum system and (b) CP specimens after applying the low-pressure MW curing.

generates MW powers of 800 W (1 magnetron) and 1,600 W (2 magnetrons) with continuous MW application. The gained compressive strength development of the MW-cured CP is evaluated by comparing the rate of compressive strength development of CP with the lime-saturated water-cured CP in the first 28 days. The additional MW curing condition is the controlled temperature in the cavity and CP specimen, which does not exceed 70 °C. Here, the bleeding effect on the compressive strength of the specimens with the surface/length ratio was not considered, and curing occurred before the initial setting to reduce the water/cement ratio, resulting in a densified paste structure.

## 2. Experimental

### 2.1. Materials

CP mixtures were prepared by mixing Portland cement Type 1 (OPC)

**Table 2**  
Mixture proportions of CP.

Water–cement ratio (w/c) (by mass)	OPC (kg/m <sup>3</sup> )	Water (kg/m <sup>3</sup> )	Water reducing admixture Type A [22] (Specific Gravity = 1.01) (kg/m <sup>3</sup> )	Assumed air content (% by volume)
0.25	1730	433	8.65	1.0
0.35	1472	515	7.36	1.0
0.45	1282	576	6.41	1.0

and tap water (pH  $\approx$  7.5). The chemical compositions and physical properties of OPC are shown in Table 1. The main composition of OPC is CaO, and the secondary contents in descending order are SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub>. The particle size of the OPC at 50% cumulative passing is 13.50  $\pm$  0.39  $\mu$ m, which is consistent with the specific surface area obtained via the Brunauer–Emmett–Teller (BET) method (852  $\pm$  14.02 m<sup>2</sup>/kg), and the specific gravity of the OPC is 3.15  $\pm$  0.02. To further increase the formability of the cement paste at a low w/c (such as 0.25), the water-reducing admixture Type A, which complies with ASTM C494-17 [22] and has a specific gravity of 1.01, was used with the recommended dosage of 0.5% wt. of OPC content.

## 2.2. Instrumentation

The multi-mode double-feed MW system with a vacuum pump is used to dewater the CP. The physical dimension and complementary equipment are shown in Fig. 1(a) [23]. The MW system consists of a cylindrical MW cavity made of metallic steel with dimensions of 24 cm in diameter and 72 cm in length in the horizontal direction (volume of MW cavity is approximately 1.30  $\times$  10<sup>5</sup> cm<sup>3</sup>) and polypropylene (PP) lining with dimensions of 22 cm in diameter and 50 cm in length (the volume of the MW cavity is approximately 0.76  $\times$  10<sup>5</sup> cm<sup>3</sup>) in the horizontal direction. The air-cooled magnetrons are installed in the roof and side of the MW cavity (here, the top magnetron is called as magnetron 1 (vertical (symmetric) direction), and the side magnetron is called magnetron 2 (horizontal (asymmetric) direction)). Each magnetron can generate MW with power of 800 W, and 2 magnetrons can generate 1,600 W and operate in the continuous MW generation mode. The third part of the MW system is the control part, which controls the rate of MW feeding of magnetrons 1, 2 and 1 and 2, and the internal temperature in the cavity during the MW application is not more than 70 °C. The last part is a vacuum pump with a power of 1.0 hp to remove the internal air from the ambient pressure (101.325 kPa) and decrease it to 50 and 30 kPa.

## 2.3. Methods

CP was prepared by determining the water content, which is controlled with the specific w/c of 0.25, 0.35 and 0.45 with compensating for water in the admixture used, and the air content in the CP mixtures was assumed at 1.0% vol. as shown in the CP mix proportions in Table 2. With the volume controlled at 1.0 m<sup>3</sup> CP and air content, the increase in the w/c ratio yields an increase in water content and a decrease in OCP content. After designing the mix proportions, we mixed the CP in accordance with ASTM C305-14 [24] by weighing the number of CP ingredients for 12 specimens for each of four mixing batches and pouring and compacting them in acrylic molds with dimensions of 5 cm long  $\times$  5 cm wide  $\times$  10 cm thick in accordance with ASTM C109/C109M - 16a [25]. Our calculations were consistent with uniform MW propagation within the cavity of this prototype, and we wrapped the molds in a plastic sheet to prevent water evaporation from the specimen. Here, the first set of 48 specimens was demolded at 6.0  $\pm$  0.5 h after mixing and water curing in lime-saturated water with the temperature controlled at 25 °C until the testing age. The compressive strength of the CP specimens at the ages of 1, 3, 7, and 28 complied with ASTM C39/C39M-18 [26].

To produce the CP, the sets of 192-CP specimens in dimensions of 5

**Table 3**  
Case studies of CP under MW curing.

Case	w/c	Pressure within cavity (kPa)	Magnetron	Number of CP specimens	Time of application (minutes)
1	0.25	30	1	12	100
2	0.25	50	1	12	100
3	0.35	30	1	12	50
4	0.35	50	1	12	40
5	0.45	30	1	12	50
6	0.45	50	1	12	40
7	0.35	30	1	24	60
8	0.35	50	1	24	60
9	0.35	50	2	12	70
10	0.35	50	1 and 2	12	30
11	0.35	50	2	24	60
12	0.35	50	1 and 2	24	40

cm  $\times$  5 cm  $\times$  10 cm of length  $\times$  width  $\times$  thickness were cast. After 30 min of mixing, before applying MW curing, the set of CP specimens per batch of MW curing (12 and 24 specimens) were continuously cured as follows:

- Step 1: arrange the CP specimens (Fig. 1(b)) in molds on the tray made with 0.6-cm-thick acrylic bars and sheets.
- Step 2: close the cavity cap and pump air out of the cavity to decrease the final internal pressure to 30 or 50 kPa and consequently to proceed with the MW curing as shown in the case studies in Table 3.
- Step 3: measure the increase in temperature after MW curing at the surface of the middle CP using an infrared camera and the moisture content of the specimens by weighing the loss weight of the CP specimen and calculating remaining w/c. The surface temperature of CP specimens and cavity are detected immediately after opening the cavity cap.
- Step 4: demold the MW-cured CP specimens and wrap them in a plastic sheet to prevent water loss. Curing is conducted until the testing ages of 1, 3, 7, and 28 days in accordance with ASTM C39/C39M-18 [26] to evaluate the CP by comparing its compressive strength to that of water-cured CP at 1, 3, 7, and 28 days.

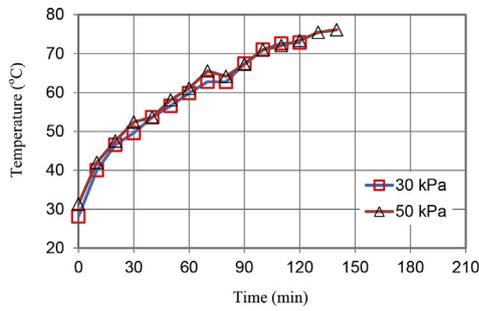
## 3. Results

To achieve curing using low-pressure MW curing in the production of CP, the effects of the pressure in the MW cavity, the feed direction of MW and the number of specimens per batch of MW curing are presented and discussed in detail.

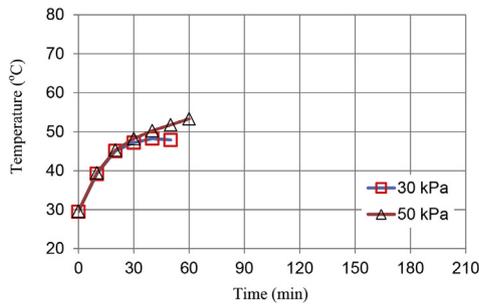
### 3.1. Effect of pressure in the MW cavity on the temperature increase and moisture content

This part examines the effect of pressure in the MW cavity on the temperature increase and moisture content (in term of remaining w/c) due to the accelerated curing technique with low-pressure MW curing and varying w/c, which is the predominant parameter to determine the rate of the hydration reaction or the rate of strength development of the CP. Curing is removing water (moisture) from the internal CP structure by the vapor pressure, which is created by heating water molecules using the MW heating mechanism. Here, the radiation direction of MW is symmetrically perpendicular to the horizontally placed CP specimens. Only magnetron 1 (vertical) was used and radiated a MW power of 800 W onto the CP specimens.

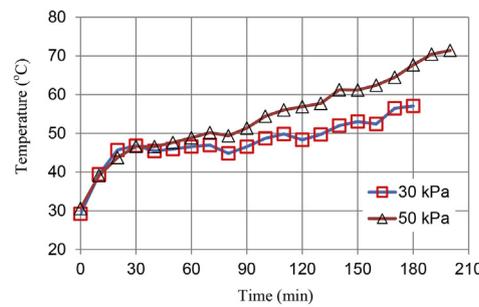
Fig. 2 shows the temperature increase in the CP specimens under pressures of 30 and 50 kPa for 12 specimens per batch of MW curing with control initial w/c values of 0.25 (cases 1 and 2), 0.35 (cases 3 and 4) and 0.45 (cases 5 and 6). In cases 1 (30 kPa) and 2 (50 kPa), in the initial stage (approximately the first 30 min) of MW curing, the temperature of the CP specimens continuously increased rapidly (0.72 °C/min for 30 kPa and



(a) 12 specimens, w/c of 0.25, pressures of 30 and 50 kPa, magnetron 1 (vertical magnetron)



(b) 12 specimens, w/c of 0.35, pressures of 30 and 50 kPa, magnetron 1 (vertical magnetron)

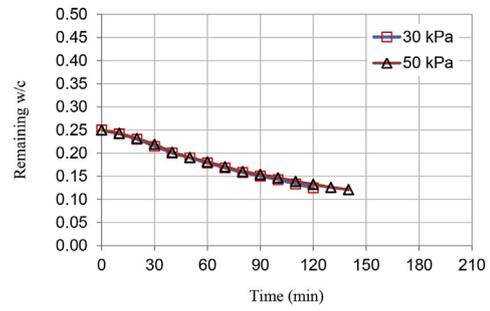


(c) 12 specimens, w/c of 0.45, pressures of 30 and 50 kPa, magnetron 1 (vertical magnetron)

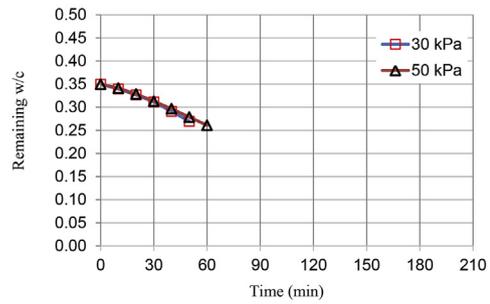
**Fig. 2.** Temperature increase and time of CP under the pressures of 30 and 50 kPa and magnetron 1 for 12 specimens with a controlled w/c of (a) 0.25, (b) 0.35 and (c) 0.45.

0.70 °C/min for 50 kPa); from 30 min to 100 min, the rate of temperature increase slowed (0.64 °C/min for 30 kPa and 0.40 °C/min for 50 kPa). This behavior is consistent with the characteristics of CP that the delay time of 30 min is the dormant period, during which the hydration reaction occurs at low rates because of the primary ettringite (Ca<sub>6</sub>Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>(OH)<sub>12</sub>·26H<sub>2</sub>O) formation. When CP is heated, the dormant period is shortened, and the hydration reaction resumes with an increasing rate as observed from the rate of temperature rise from 30 min to 60 min. After 100 min of MW curing, the temperature increased because of the heat generation from the MW and hydration reaction. The maximum temperature increase at the end of curing (100 min application time) of cases 1 and 2 are similar (~71 °C). This shortening of the dormant period of the CP may cause the secondary delayed ettringite formation (which causes cracking under moisture repletion) due to the sedimentation of primary ettringite at high temperature (70–90 °C). Thus, the highest temperature increase of CP subjected to MW curing cannot exceed 70 °C in this study. Case 1 of CP with a low w/c of 0.25 may have delayed ettringite formation because the internal maximum temperature was more than 70 °C.

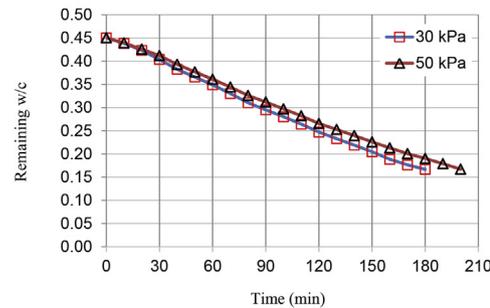
When comparing the effect of the pressure level in the MW cavity on the temperature increase, the temperature hardly varied among CP specimens at the pressure levels of 30 and 50 kPa because at low w/c, the low amount of water cannot transfer and evaporate from the CP specimen.



(a) 12 specimens, w/c of 0.25, pressures of 30 and 50 kPa, magnetron 1 (vertical magnetron)



(b) 12 specimens, w/c of 0.35, pressures of 30 and 50 kPa, magnetron 1 (vertical magnetron)



(c) 12 specimens, w/c of 0.45, pressures of 30 and 50 kPa, magnetron 1 (vertical magnetron)

**Fig. 3.** Moisture content and time of CP under the pressures of 30 and 50 kPa and magnetron 1 (vertical magnetron) for 12 specimens with a controlled w/c of (a) 0.25, (b) 0.35 and (c) 0.45.

In Fig. 2(b), for cases 3 and 4, the initial w/c increased from 0.25 to 0.35, and the application times of MW curing was 50 min (case 3) and 40 min (case 4). The rates of temperature increase in the first 30 min of low-pressure MW curing are 0.59 °C/min for 30 kPa and 0.62 °C/min for 50 kPa similar to the data from Makul et al. [23], however this study extended the range of experiments in order to consider the continuity of the rate of temperature increase at low remaining w/c. In these cases, unlike cases 1 and 2, the increasing amount of water affects the amount of heat generation by MW, the amount of absorbed heat and the low rate of the hydration reaction (the high water content causes the low formation rate of calcium-silicate-hydrate (C–S–H)). At the end of the MW curing process, there is a difference in CP specimen temperature between the pressures of 30 and 50 kPa (difference at 100 min = 3.90 °C) compared to cases 1 and 2 (difference at 100 min = 0.10 °C), which indicates that w/c affects to the rate of temperature increase of the specimen and the difference in temperature at the end of the MW curing, and the temperature difference increases when the w/c ratio increases.

Fig. 2(c) shows cases 5 and 6 of CP prepared by controlling the w/c ratio of 0.45 with application time of 50 min (case 5) and 40 min (case 6). The temperature increase is similar to that in cases 3–4 and the data from Makul et al. [23]. The rates of temperature increase in the first 30 min of MW vacuum are 0.59 °C/min for 30 kPa and 0.54 °C/min for 50 kPa. This behavior can be described as compensating effects from the heat

generation, i.e., a large amount of heat can be generated from a high water content, which has a high dielectric constant [27]; in contrast, the hydration reaction rate is low because of the low formation rate of C–S–H. Hence, the maximum temperature increases at the end of curing of cases 5 ( $\sim 47.6^\circ\text{C}$ ) and 6 ( $\sim 46.8^\circ\text{C}$ ) are similar.

As mentioned, the curing of CP relates the temperature increase from low-pressure MW curing (heating up) and heat from the hydration reaction with outward moisture transfer. Fig. 3 shows the moisture content (in terms of the remaining w/c) and time at pressures of 30 and 50 kPa with magnetron 1 (vertical) for 12 specimens at a controlled initial w/c of 0.25, 0.35 and 0.45. The remaining w/c of CP continuously decreased with more MW curing application time, and the final w/c of case 1 after low pressures at 30 and 50 kPa (0.14) is close to that of case 2 (0.15), which can improve the early-age compressive strength of CP (1 day after MW curing) but reduce the compressive strength of CP at 7 and 28 days because the minimum required w/c level of complete hydration reaction should be 0.22 (stoichiometry-based calculation) and even lower than that of the real completion of hydration reaction of pore water at 0.38. Therefore, the application time of MW curing for cases 1 and 2 should not exceed 30 min. The average rate of w/c remaining until 100 min of case 1 ( $1.07 \times 10^{-3}$  per min) is similar to that in case 2 ( $1.00 \times 10^{-3}$  per min), which indicates that the low w/c CP at a pressure of 30 kPa does not affect the temperature increase compared to case 2 at 50 kPa with a small amount of water from the CP specimens.

When w/c increases to 0.35 (Fig. 3(b)), the rates of w/c remaining in both cases of 30 and 50 kPa are higher in case 3 ( $1.61 \times 10^{-3}$  per min) than in case 4 ( $1.33 \times 10^{-3}$  per min). This difference indicates that at lower pressure, the internal water of CP can be quickly removed and evaporated from the specimens because the difference in vapor pressure in the specimens is higher than the pressure in the cavity. This behavior is similar in cases 5 ( $1.69 \times 10^{-3}$  per min) and 6 ( $1.43 \times 10^{-3}$  per min) with 0.45-w/c CP as shown in Fig. 3 (c).

As shown in Table 2, OPC content and moisture (water) content each has a significant effect on the rate at which the temperature increases and the moisture decreases when CP is cured by MW. For example, for CP subjected to MW at a power of 800 W the 1 magnetron, pressure levels of 30 and 50 kPa, at 0.25-w/c CP (OPC content at  $1,730\text{ kg/m}^3$  and water content of  $433\text{ kg/m}^3$ ), the temperature increased continuously at the highest rate of all the specimens in the study due to the high hydration reaction rate from the high OPC content's reaction with water. Through this reaction, exothermic heat is produced, which liberates the C–S–H formation associated with the dielectric value of the water being higher than the dielectric value of the OPC. Free water can absorb microwave energy and convert it into additional heat, which results in a decrease in the moisture content and gains in high compressive strength. Increasing the w/c to 0.35 (OPC content of  $1,472\text{ kg/m}^3$  and water content of  $515\text{ kg/m}^3$ ) has the result of generating significant additional heat from the MW. However, cement content seems to decrease in relation to the heat from the CP hydration, resulting in higher remaining moisture content than is the case with 0.25-w/c CP, which gives rise to gain in compressive strength. This effect is clear in the case of 0.45-w/c CP (OPC content of  $1,282\text{ kg/m}^3$  and water content of  $576\text{ kg/m}^3$ ) such that compared to the rest of the samples, this CP has the lowest temperature increase and highest remaining moisture content, which leads to the lowest compressive strength.

### 3.2. Effect of the feed direction of MW on the temperature increase and moisture content

Fig. 4 shows the temperature increase and time of CP prepared at an initial w/c of 0.35 and pressures of 30 kPa (Fig. 4(a)) and 50 kPa (Fig. 4(b)) using magnetron 1 (vertical (symmetric in vertical direction), magnetron 2 (horizontal (asymmetric in vertical direction) magnetron) and both magnetrons 1 and 2 for 12 CP specimens. In Fig. 4(a), the trends of temperature increase are similar among the cases with magnetron 1 (case 3), magnetron 2 (case 7), and magnetrons 1 and 2 (case 10). The

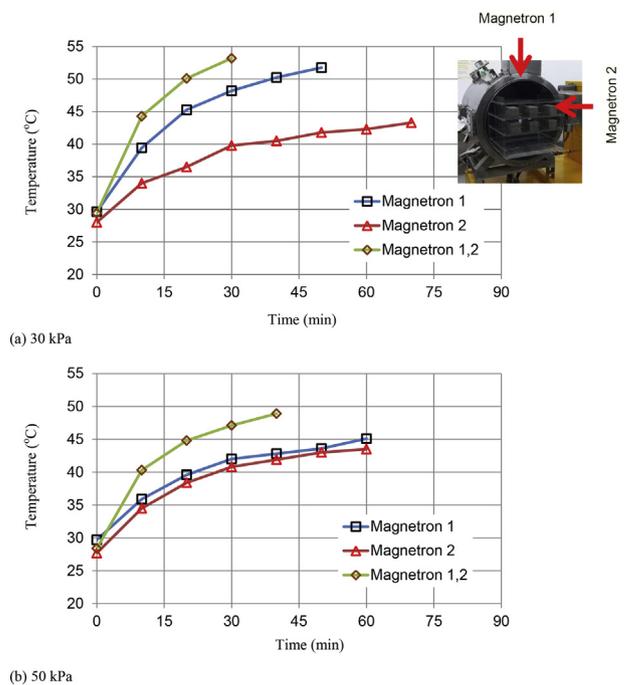


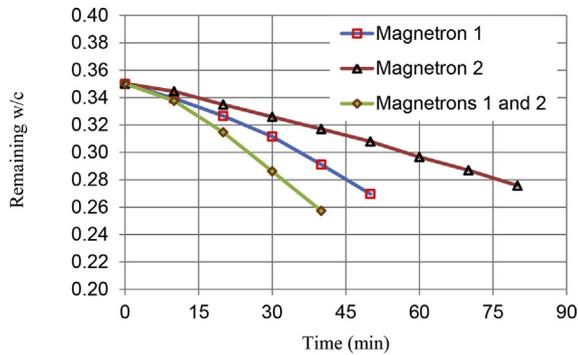
Fig. 4. Temperature and time of CP with an initial w/c of 0.35 and pressures of 30 and 50 kPa at magnetron 1 (vertical) magnetron 2 (horizontal) magnetron, and both 1 and 2 for 12 specimens of (a) 30 kPa and (b) 50 kPa.

increasing temperature caused by heat generated by MW curing in the dormant period with hydration reaction proceeds with a low rate or is almost stationary. Thus, heat is mainly generated from the MW heating, and in this period, the CP specimen has water, which can absorb more MW energy and convert into heat. After 30 min of MW curing, the rate of temperature increase has slowed to almost a constant because a low amount of water interacts with MW, and there is a change in state from free water to combined water of the C–S–H structure.

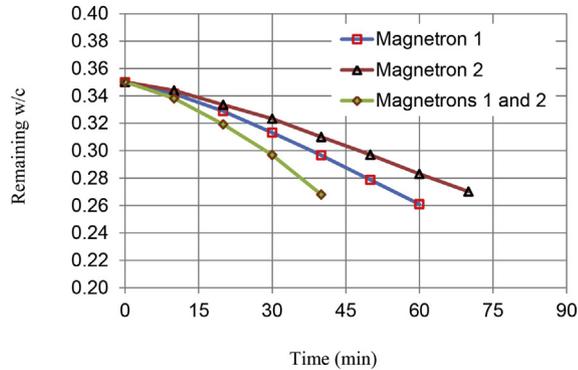
However, there are many differences in the rate of temperature increase. The average rate of temperature increase of magnetron 1 in the first 30 min of MW curing at a power of 800 W is  $0.77^\circ\text{C}/\text{min}$ , whereas that of magnetron 2 (800 W) is  $0.61^\circ\text{C}/\text{min}$  and that of 2 magnetrons (1,600 W) is  $0.32^\circ\text{C}/\text{min}$ . To compare the same power (magnetrons 1 and 2), the difference in temperature increase causes the magnetron placement, i.e., 1 is symmetrically perpendicular with the horizontal position of the specimens, so all specimens absorb similar amounts of MW energy, i.e., the temperature can steadily increase throughout all specimens. With magnetron 2, the magnetron is placed asymmetrically, which causes a nonuniform MW distribution. Thus, the specimen near the magnetron receives more MW energy earlier, and the MW distribution in the cavity induces hot spots at some locations, which results in a nonuniform heat distribution. In case 10, with the use of magnetrons 1 and 2 (total power at 1,600 W), due to the high MW power (2 times compared to magnetron 1 or 2), the rate of temperature increase is almost 20% and 30% higher than that with magnetron 1 and magnetron 2 individually. The rate of temperature increase is close to the rate of temperature from magnetron 1, so the MW power generated by magnetron 1 has a stronger effect than the heat generation from magnetron 1. However, the use of two magnetrons conversely creates more hot spots in the CP specimens, which affects the quality of the internal structure of CP.

For cases 4, 8 and 12 with a pressure in the cavity of 50 kPa (Fig. 4(b)), the trends of temperature increase are similar, but the rates are lower than that of 30 kPa. This low rate of temperature increase may result from the state change of water: at high pressure, water converts to vapor and is more easily removed.

In Fig. 4(b), the rate of temperature change using magnetron 1 is close



(a) 30 kPa



(b) 50 kPa

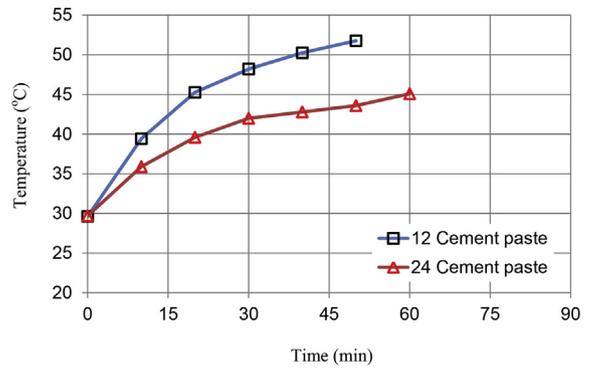
Fig. 5. Moisture content and time of MW curing with an initial w/c of 0.35 and a pressure of 50 kPa, magnetron 1 (vertical magnetron), 2 (horizontal magnetron), and both 1 and 2 for 12 specimens of (a) 30 kPa and (b) 50 kPa.

to that of using magnetron 2, which is different from the case at the pressure of 30 kPa. This phenomenon can imply that at high pressures, the magnetron configuration of symmetry and asymmetry has no effect because of the more similar MW distributions between magnetrons 1 and 2.

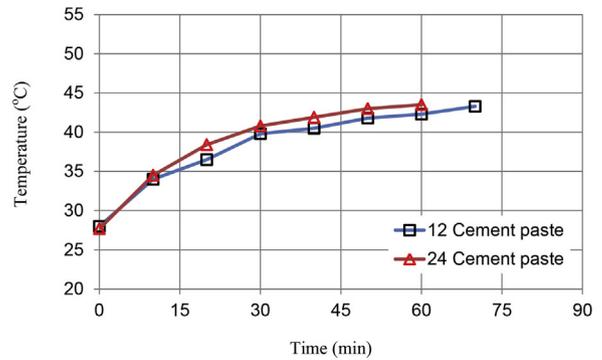
Fig. 5 presents the decrease in the remaining w/c of CP for the cases with magnetrons 1, 2 and 1 and 2 at pressures of 30 kPa (Fig. 5 (a) (cases 3, 6 and 10)) and 50 kPa (Fig. 5 (b) (cases 4, 8 and 12)). Both pressure have similar trends of decreasing w/c. The MW curing using 2 magnetrons (power of 1,600 W) at 30 min MW application have the highest rate ( $2.0 \times 10^{-3}$  per min), the second is the case with magnetron 1 ( $1.33 \times 10^{-3}$  per min), and the last is the case with magnetron 2 ( $0.67 \times 10^{-3}$  per min). These results are consistent with the increase in temperature in Fig. 4, which is related to the change in water from free water to fixed water in CP specimens and the formation of C-S-H. In detail, with magnetron 2, the heat generation from MW curing induces the highest temperature in the specimen and continuously affects the high C-S-H formation rate more than the case with magnetron 1 or 2.

Comparing the rate of decrease in w/c for the case of a pressure of 50 kPa, we find that using magnetron 1, 2, and 1 and 2 is similar to the case of pressure 30 kPa. At both pressures of 30 and 50 kPa, there is little difference, i.e., neither pressure substantially affects the amount of water that is forced to the vapor state and removed from the internal specimen. Thus, most of the high-temperature water in the CP structure is heated and positively affects to increase the rate of hydration reaction with the low amount of loss water and develop the compressive strength of CP.

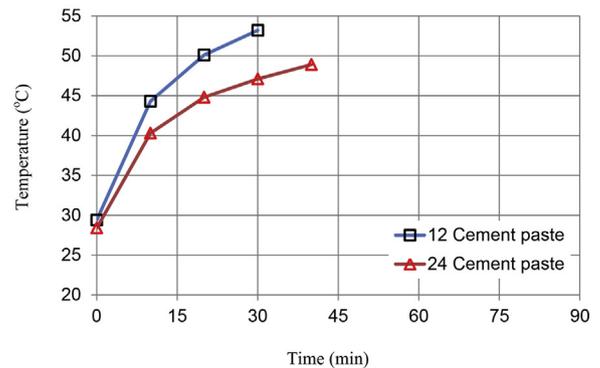
One point of observation is the final w/c of the CP after the MW curing. Based on the stoichiometry-based calculation, w/c should be more than 0.22 to ensure the perfect hydration reaction. Based on Fig. 5 (a), compared with the w/c of 0.22, the time of MW application times may extend to 75 min, 150 min and 45 min for using magnetron 1,



(a) Magnetron 1 (vertical magnetron)



(b) Magnetron 2 (horizontal magnetron)



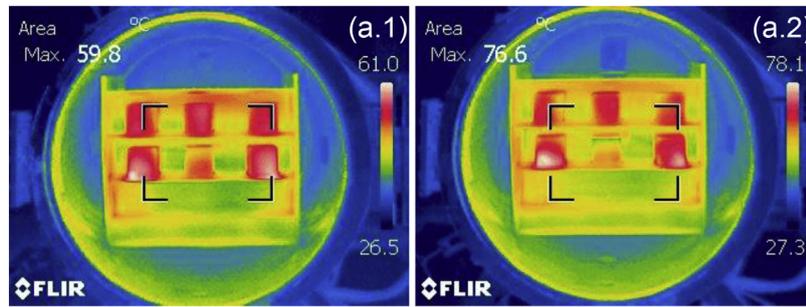
(c) Magnetrons 1 (vertical magnetron) and 2 (horizontal magnetron)

Fig. 6. Temperature and application time of CP under a pressure of 50 kPa with a controlled initial w/c of 0.35 and 12 and 24 specimens using (a) magnetron 1 (vertical magnetron) (b) magnetron 2 (horizontal magnetron) and (c) magnetrons 1 and 2.

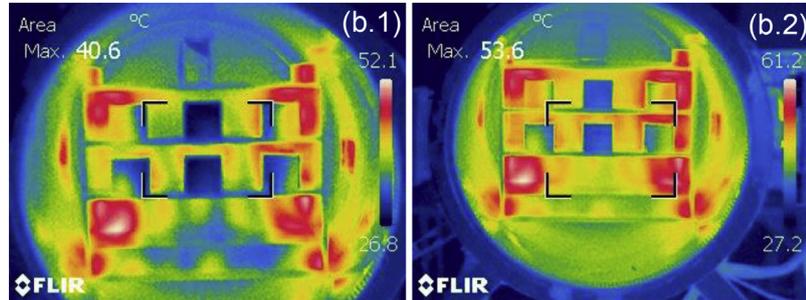
magnetron 2, and magnetrons 1 and 2. However, for the case in Fig. 5(b) at a pressure of 50 kPa, the application time should be 75 min, 90 min and 45 min for using magnetron 1, magnetron 2, and magnetrons 1 and 2.

### 3.3. Effect of the number of CP specimens on the temperature increase and moisture content

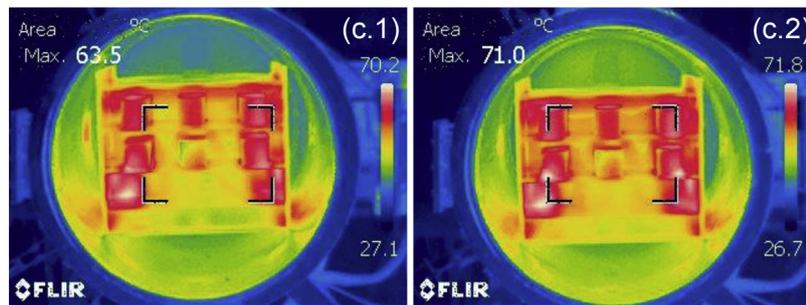
Fig. 6 shows the temperature and application time of CP subjected to a pressure of 50 kPa with a controlled initial w/c of 0.35 and 12 (case 4) and 24 (case 10) specimens per batch using magnetron 1 (vertical magnetron), magnetron 2 (horizontal magnetron), and magnetron 1 and 2. Both cases of 12 and 24 specimens have similar trends of temperature increase. In the initial stage of MW curing (first 30 min of MW curing), the rates of temperature increase of CP specimens are high and decrease



(a) Distribution of the surface temperature of CP at a pressure of 50 kPa, w/c of 0.35, magnetron 1, and 12 specimens at (a.1) 20 min and (a.2) 40 min



(b) Distribution of the surface temperature of CP at a pressure of 50 kPa, w/c of 0.35, magnetron 2, and 12 specimens at (b.1) 20 min and (b.2) 40 min



(c) Distribution of the surface temperature of CP at a pressure of 50 kPa, w/c of 0.35, magnetrons 1 and 2, and 12 specimens at (c.1) 20 min and (c.2) 40 min

Fig. 7. Distribution of the surface temperature of CP and cavity at a pressure of 50 kPa with an initial w/c of 0.35 and 12 specimens at 20 and 40 min using the feed direction of (a) magnetron 1 (vertical magnetron), (b) magnetron 2 (horizontal magnetron), and (c) magnetrons 1 and 2.

to almost constants. For example, in Fig. 6(a), with magnetron 1 (symmetric MW radiation), the rate of temperature increase in the first 30 min of MW curing of 12 and 24 specimens is 0.61 °C/min and 0.40 °C/min. After 30 min, the rate is 0.18 °C/min and 0.10 °C/min for 12 and 24 specimens, respectively. This characteristic temperature increase can be described as follows: for the same amount of energy, 24 specimens per batch gain less energy per specimen than 12 specimens per batch.

In the case with magnetron 2 (cases 9 and 11), the difference in temperature increase between 12 and 24 specimens was very small. On average, in the first 30 min of MW application, 12 specimens have 0.38 °C/min, whereas 24 specimens have 0.36 °C/min because the MW distribution of 12 and 24 specimens per batch barely changes the heat generation, since the MW is identical.

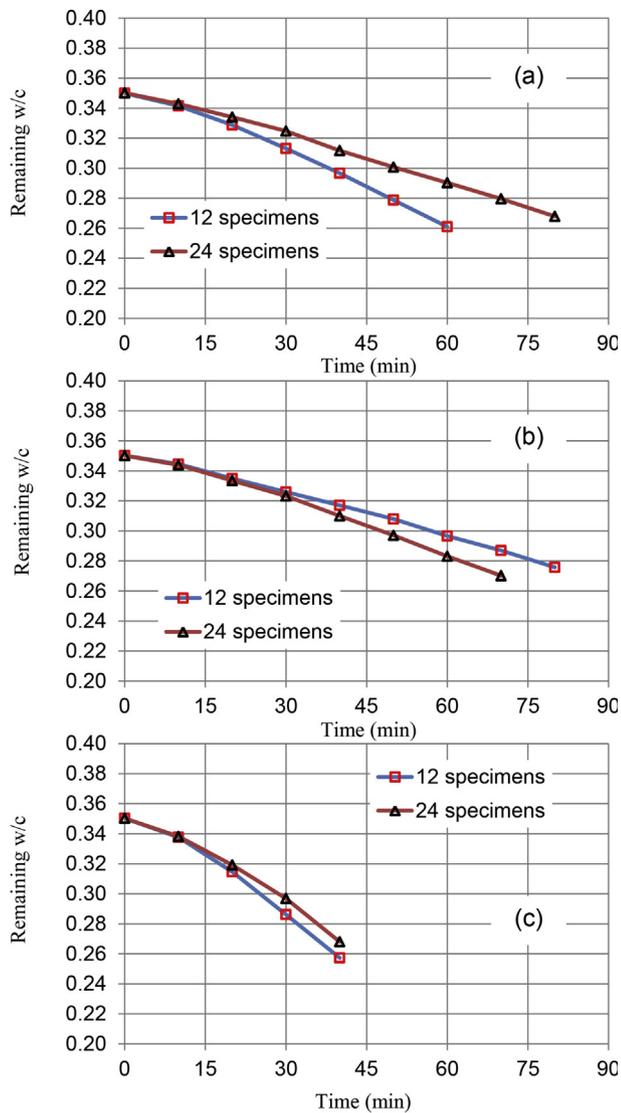
In the case with magnetrons 1 and 2 (1,600 W power), the temperature increase is similar to the case with magnetron 1: the rate is higher with 12 specimens per batch than with 24 specimens per batch, which shows that magnetron 1 is more effective than magnetron 2. Fig. 7 shows the distribution of surface temperature at a pressure of 50 kPa with an initial w/c of 0.35 and 12 specimens at 20 and 40 min using the feed

direction of magnetron 1 (vertical magnetron), magnetron 2 (horizontal magnetron), and magnetrons 1 and 2. In case 10, when magnetron 1 is used, the middle specimens are heated (Fig. 7(a)); however, when magnetron 2 is used, the specimens have low temperature increase (Fig. 7(b)) because the more uniform MW distribution in the specimens with magnetron 1 results in higher heat than those with magnetron 2.

As shown in Fig. 8, the decrease in remaining w/c is consistent with the increase in temperature, which can be calculated as the rate of decrease in w/c. For example, the rate of decrease in temperature w/c of 12 specimens with magnetron 1 in the first 30 min of MW curing is  $1.33 \times 10^{-3}$  per min. The reason is that the high amount of heat generated by MW curing converts more water in the specimen to vapor and squeezes water from the CP specimens.

### 3.4. The gained compressive strength of CP

The compressive strength gained when CP is subjected to MW curing depends on the temperature increase and the moisture content (remaining w/c). Fig. 9 shows the compressive strength for days 1–28 of



**Fig. 8.** Moisture content (remaining w/c) and application time of CP under the pressure of 50 kPa with an initial w/c of 0.35 and 12 and 24 specimens using the feed direction of (a) magnetron 1, (b) magnetron 2, and (c) magnetrons 1 and 2.

testing with an initial w/c ratio of 0.25 (Fig. 9(a)), 0.35 (Fig. 9(b)), and 0.45 (Fig. 9(c)) at 30 kPa for magnetron 1 and 12 specimens. Fig. 9(d) shows the CP with an initial w/c ratio of 0.35 at 50 kPa with magnetron 1 and 24 specimens. The CP samples under low-pressure MW curing and water curing for both 12 and 24 specimens per batch developed compressive strength throughout the 28-day period. Compared on the basis of the same initial w/c, the MW-cured CP specimens under a pressure of 30 (case 1) and 50 kPa (case 2) had higher compressive strength than did the water-cured CP. For example, at a w/c of 0.35, the 1-day strength of the MW-cured CP specimens subjected to 30 kPa and 50 kPa were 42.3% and 48.2% higher, respectively, than that of the water-cured CP specimens. The reason is that curing CP via MW in the dormant period has the effect of shortening this period and increases the hydration rate, which increases the amount of C–S–H such that that CP develops a high level of compressive strength [28, 29]. Under MW curing, the CP samples with a low pressure of 50 kPa and 30 kPa showed similar compressive strength.

At 1 day after MW curing, the CP with the initial w/c of 0.35 can develop higher compressive strength than the water-cured sample but lower than the 0.25-w/c paste because the 0.35-w/c sample has more free water than the 0.25-w/c sample which implies more pores, lower

internal structure density, and lower hydration reaction rate. This behavior is similar when w/c increases to 0.45. Moreover, after day 1 (at 3, 7, and 28 days), the MW-cured CP also developed greater compressive strength than did the water-cured paste. However, compressive strength developed more slowly than was the case at day 1. Thus, the low-pressure cavity associated with MW curing can solve the problem of high-temperature curing by maintaining the maximum temperature at below 70 °C [29, 30].

#### 4. Discussion

The energy consumptions (power-associated application time of MW curing) of the CP are shown in Table 4. The moisture content as the remaining w/c of 0.25- w/c CP decreases from 0.25 to 0.13, that of 0.35-w/c CP decreases to 0.26, and that of 0.45-w/c CP decreases to 0.18. For cases 1–6, the CP (12 specimens and pressure at 30 kPa) with 0.25 (cases 1 and 2) consumes a larger power-time ratio than those with 0.35 (cases 3 and 4) and 0.45 (cases 5 and 6). The reason is that above 0.25, water is classified as free water in the capillary pore, and free water can be easily forced to move outward. In contrast, at low w/c (less than 0.25), water is combined as a part of the calcium-silicate-hydrate structure and is more difficult to force outward. In the case of the 0.35-w/c CP (cases 3 and 4) and 0.45-w/c CP (cases 5 and 6), the energy consumption has similar values, indicating that it does not vary during MW curing.

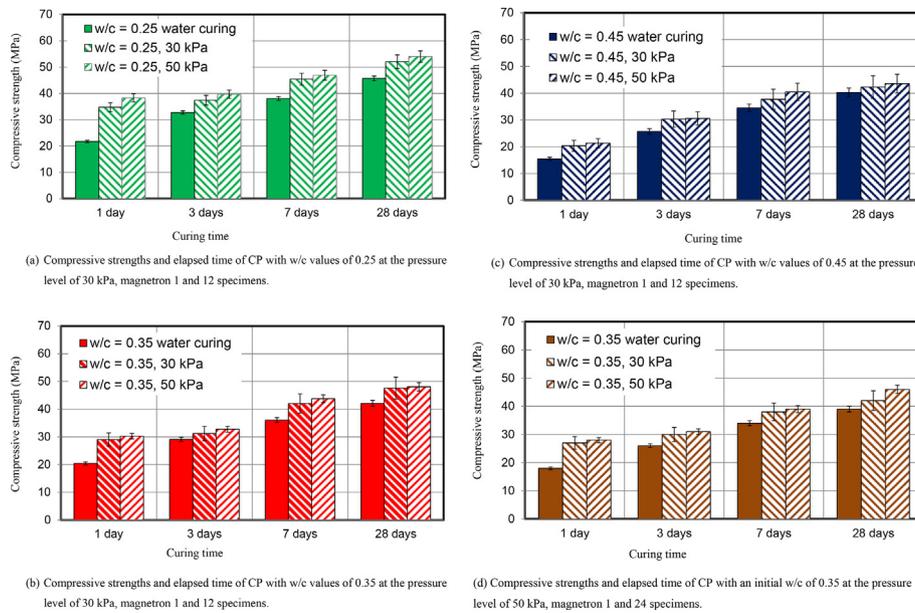
By applying magnetrons 1 and 2 to the 12 CP specimens (case 10), more energy is consumed than in the case with only magnetron 2 (case 9), a result which shows that the use of magnetron positions 1 (vertical magnetron) and 2 has lower efficiency compared to the use of magnetron 2 alone (unsymmetrical magnetron) because of the destruction of the electric MW field and the occurrence of hot spots. This behavior is comparable to those in case 11 (magnetron 2) and case 12 (vertical and horizontal magnetrons).

For the suitable MW curing cases, there are two options to minimize the energy consumptions: controlling the 0.35- and 0.45- w/c CP at a pressure of 30 kPa (case 3) for 50 min and controlling 12 specimens at 0.45 (case 5) for 120 min. The case of 0.25-w/c for 12 specimens is not presented because decreasing w/c to 0.25 w/c had a destructive effect on the compressive strength of the CP.

#### 5. Conclusions

Based on the experimental results, the following conclusions can be drawn:

- The successful use of low-pressure MW heating and curing to produce high-early-compressive-strength CP must be considered in relation to the characteristics of CP, specifically the w/c, as well as the delay time before the MW is applied and the method through which the specimen is prepared. Additionally, the MW equipment and processing (the pressure in the MW cavity, the sequence of the MW curing process, the number of specimens, and the feed direction of the MW propagation) must be taken into account.
- When certain conditions are created—i.e., w/c (0.25, 0.35, and 0.45), MW power (800 and 1600 W) and pressure level (30 and 50 kPa)—the temperature of the CP increases continuously at a high rate. Then, the rate at which the temperature increases shows a slight decrease consistent with the moisture content of the CP.
- To compare the samples CP, two magnetrons were used: one was placed symmetrically perpendicular to the horizontal position of the specimens (magnetron 1) and higher than the position of the asymmetrically placed magnetron (magnetron 2), thereby avoiding nonuniform MW propagation and nonuniform heat distribution. Here, a symmetric magnetron is suitable for MW curing in order to achieve the highest compressive strength.
- The cases of MW curing with 12 specimens inserted in the cavity (3.95% of the volume of the microwave cavity) and 24 specimens



**Fig. 9.** Compressive strengths of (a) CP with w/c values of 0.25 at the pressure level of 30 kPa, magnetron 1 and 12 specimens, (b) CP with w/c values of 0.35 at the pressure level of 30 kPa, magnetron 1 and 12 specimens, (c) CP with w/c values of 0.45 at the pressure level of 30 kPa, magnetron 1 and 12 specimens and (d) CP with an initial w/c of 0.35 at the pressure level of 50 kPa, magnetron 1 and 24 specimens.

**Table 4**  
Energy consumption of CP for MW-assisted curing.

Case	Parameters and conditions	Time (min)	Power (kW.hour)	Power/time (kW)
1	w/c of 0.25, pressure of 30 kPa, magnetron position 1 (vertical magnetron), 12 specimens	100	4.50	2.70
2	w/c of 0.25, pressure of 50 kPa, magnetron position 1 (vertical magnetron), 12 specimens	100	4.90	2.94
3	w/c of 0.35, pressure of 30 kPa, magnetron position 1 (vertical magnetron), 12 specimens	50	2.2	2.64
4	w/c of 0.35, pressure of 50 kPa, magnetron position 1 (vertical magnetron), 12 specimens	40	2.0	3.00
5	w/c of 0.45, pressure of 30 kPa, magnetron position 1 (vertical magnetron), 12 specimens	50	2.2	2.64
6	w/c of 0.45, pressure of 50 kPa, magnetron position 1 (vertical magnetron), 12 specimens	40	2.0	3.00
7	w/c of 0.35, pressure of 30 kPa, magnetron position 1 (vertical magnetron), 24 specimens	60	2.7	2.70
8	w/c of 0.35, pressure of 50 kPa, magnetron position 1 (vertical magnetron), 24 specimens	60	3.0	3.00
9	w/c of 0.35, pressure of 50 kPa, magnetron position 2 (horizontal magnetron), 12 specimens	70	3.7	3.17
10	w/c of 0.35, pressure of 50 kPa, magnetron positions 1 (vertical magnetron) and 2 (horizontal magnetron), 12 specimens	30	1.9	3.80
11	w/c of 0.35, pressure of 50 kPa, magnetron position 2 (horizontal magnetron), 24 specimen	60	2.9	2.90
12	w/c of 0.35, pressure of 50 kPa, magnetron positions 1 (vertical magnetron) and 2 (horizontal magnetron), 24 specimens	40	2.6	3.90

inserted (7.90% of the volume of microwave cavity) behave comparably in regard to the increase in temperature increase and decrease in moisture manifested. Especially during the first 30 min of MW curing, the CP specimens showed a significant increase in temperature.

- The optimal point of CP subjected to low-pressure MW heating and curing is the point at which the OPC content is balanced with the water content due to heat from the hydration reaction and additional heat from MW. The highest temperature should not exceed 70 °C with w/c not exceeding 0.45.

**Declarations**

*Author contribution statement*

Natt Makul: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

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*Competing interest statement*

The authors declare no conflict of interest.

*Additional information*

No additional information is available for this paper.

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