APPLICATION OF A MICROWAVE OVEN TO SINTER CLAY PELLETS AS PLANTING MATERIALS

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Abstract

This study investigates the application of microwave hybrid heating (MSH) for sintering clay pellets, focusing on its configuration and effects compared to conventional heating methods. The research identifies optimal conditions for microwave hybrid heating, achieving a heating rate of 62°C/min using two plates of SiC susceptors at 100% microwave power. X-ray diffraction analysis demonstrates that phase composition remains consistent across samples sintered via both methods, indicating minimal impact of sintering temperature on phase transformations. However, significant differences emerge in physical and mechanical properties. Clay pellets sintered with MSH exhibit higher bulk density and reduced porosity and water absorption compared to conventionally sintered pellets, aligning closely with commercial products. Mechanical strength of pellets sintered conventionally shows minimal variation across temperatures, whereas MSH-treated pellets exhibit a marked increase in crushing strength, reaching 3.78 MPa at 900°C. The findings highlight MSH as a promising alternative for enhancing sintering efficiency, offering substantial improvements in mechanical properties and heating rates, thereby potentially reducing time and energy consumption in ceramic processing applications.

Keywords: Microwave hybrid heating, silicon carbide, susceptor, physical properties, mechanical properties

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Chapter 1

Introduction

1.1 Research motivation

Indoor planting has become more popular during the emergence of the Covid-19 pandemic because it provides a connection to nature and helps people relieve stress from work-from-home or home isolation. Planting materials such as clay pellets, often known as LECA or popper, are also in high demand. The sintered clay pellets have high porosity which allows them to absorb water and nutrients for the plant. The clay pellets can be used to spread over the soil to prevent the soil from losing moisture and to inhibit the occurrence of weeds on the surface of the soil. In general, the basic raw materials of the ceramic pellets include clay, glass frit, and biomass (as a pore former) and it is necessary to sinter at high temperatures of about 1200°C in conventional furnace, which takes time and a lot of energy consumed.

Microwave processing is an alternative method to sinter clay pellets. Microwave processing of materials offers rapid heating rate and saving time while being clean and environmentally friendly. Microwave heating also contributes to the enhancing of the properties of clay pellets because the material is heated by a unique process called volumetric heating. To sinter clay pellets, the microwave hybrid heating which includes the susceptor that can absorb microwave and convert it to heat, can be used to provide a high sintering temperature and create uniform heating for the ceramic pellets.

In this study, pellet clay and perlite were used as raw materials for producing clay pellet. Perlite is an amorphous volcanic glass which consists of 70–75% silicon dioxide (SiO_2). When perlite is heated at 850–900°C, it expands to form glassy bubble structure that have a remarkably high porosity. Addition of perlite into ceramics pellet can improve physical properties such as light weight, high porosity, and high water absorption, all of which are necessary features for planting materials. Although higher porosity is better for absorbing water, it also reduces the strength of clay pellet. Therefore, the sintering conditions for clay pellets should be optimized.

This research aims to develop a microwave hybrid heating system that uses a microwave oven to sinter ceramic pellet. The silicon carbide (SiC) plates are used as susceptor for microwave absorbers and generate heat. The highly microwave transparent insulator was designed and used to maintain the heat in microwave oven. The effects of microwave hybrid heating on the properties of clay pellets were investigated, and the results were compared to conventional sintering using an electric furnace. This study described the mechanism of microwave hybrid heating system, which could be a more energy-efficient method of material processing at high temperature.

1.2 Objectives of the study

1) Investigate parameters for microwave hybrid heating system, i.e., microwave power, number of susceptor, susceptor layout.
2) Study effect of sintering temperature on physical and mechanical properties of clay pellets.
3) Comparison of microwave heating and conventional heating on physical and mechanical properties of clay pellet.

1.3 Scopes of the study

This research focuses on the microwave heating of clay pellet. The main raw materials in this research were red clay and perlite. For comparison, the sintering process was conducted using microwave and conventional heating.

1) The parameters for microwave hybrid heating system were investigated.
    - The sintering temperature of sintering: 700, 750, 800, 850 and 900°C.
    - The microwave power: 240–1200 watts.
    - The number of susceptor: 1–3 plates.
2) The physical properties (bulk density, porosity, and water absorption), phase investigation, and mechanical strength of sintered ceramic pellets were investigated.
3) The effects of microwave heating and conventional heating on the properties of ceramics pellets were compared.

1.4 Benefits of the study

Microwave heating saves time and energy while improving the physical and mechanical properties of ceramics pellets when compared to conventional heating. This research could be utilized to develop environmentally friendly and time-saving techniques to fabricate ceramics pellet as planting materials.

Chapter 2

Theory and literature reviews

2.1 Fundamental of microwave

A Microwave is an electromagnetic wave that consists of an electric and magnetic field oscillating perpendicular to each other. Microwave frequencies are in the range of 300 MHz to 300 GHz and corresponding wavelengths are in the range of 1 mm to 1 m [1]. Figure 2.1 illustrates an electromagnetic wave spectrum, and the expansion view of the microwave frequency bands. Microwaves have been used in different ways and various applications. Microwaves were originally utilized in communications, and they are currently applied in many wireless communications such as satellite communications, radar signals, mobile phones, and navigation systems.

In 1950, microwave energy was firstly shown that it can be used for drying materials. Later in 1970, microwaves were used for sintering applications. Nowadays, microwaves have shown their potential to be used for processing various kinds of material such as polymers, ceramics, metals as well as composite materials [2].

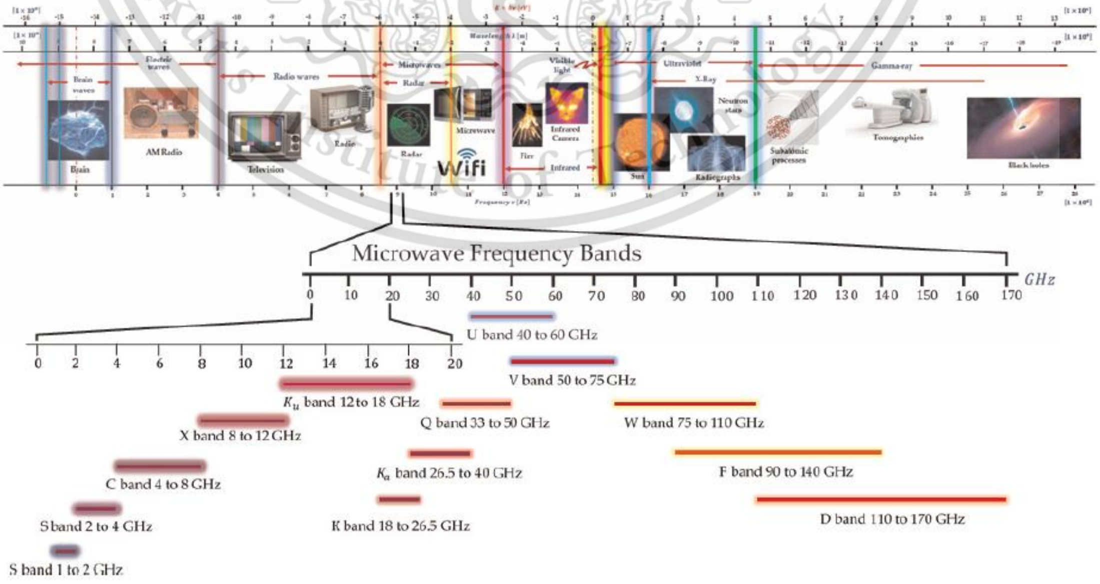


Figure 2.1 Electromagnetic wave spectrums [3].

2.2 Microwave interaction with materials

Microwave energy can be converted to heat energy depending on the type of materials that interact with it. The heat energy produced by material is determined by the quantity of energy absorbed per unit volume, which represents an amount of energy that can store and convert to heat. Based on the ability to absorb microwave energy, materials can be classified into 4 groups [2].

2.2.1 Transparent materials

Transparent materials have a remarkably high microwave penetration depth, but low microwave energy can be absorbed. It implied that almost no heat could be generated by this material when exposed to microwave. The characteristics of microwave absorption and penetration in the transparent materials are shown as category-1 in Figure 2.2. The example of microwave transparent materials are Teflon and quartz.

2.2.2 Absorber materials

This absorber materials could absorb all penetrated microwave and convert then to heat energy. Consequencely, the microwave penetration depth is gradually decreased. The microwave absorption and penetration behavior in the absorber materials are shown as category-2 in Figure 2.2. Water and SiC are examples of particular good absorber materials.

2.2.3 Opaque materials

Opaque material such as highly conductive metal has extremely low penetration depth in which microwave get almost totally reflected or slightly absorbed. So, heat is difficult to occur in this type of material during exposure to microwave. The characteristics of microwave absorption and penetration in the opaque materials are shown as category-3 in Figure 2.2. This group includes all metals in rigid bulk.

2.2.4 Mixed absorbers/composites

Mixed absorbers are the composites which contain at least one of the absorber materials in the composition. When exposed to microwave radiation, the absorber change microwave to heat energy allowing other phases in composite are being heated by microwave. The microwave absorption and penetration behavior in the mixed absorbers are similar to that of absorber materials (category-2) as shown in Figure 2.2. The example of materials in this group includes polymer matrix composites (PMC), ceramic matrix composites (CMC), and metal matrix composites (MMC).

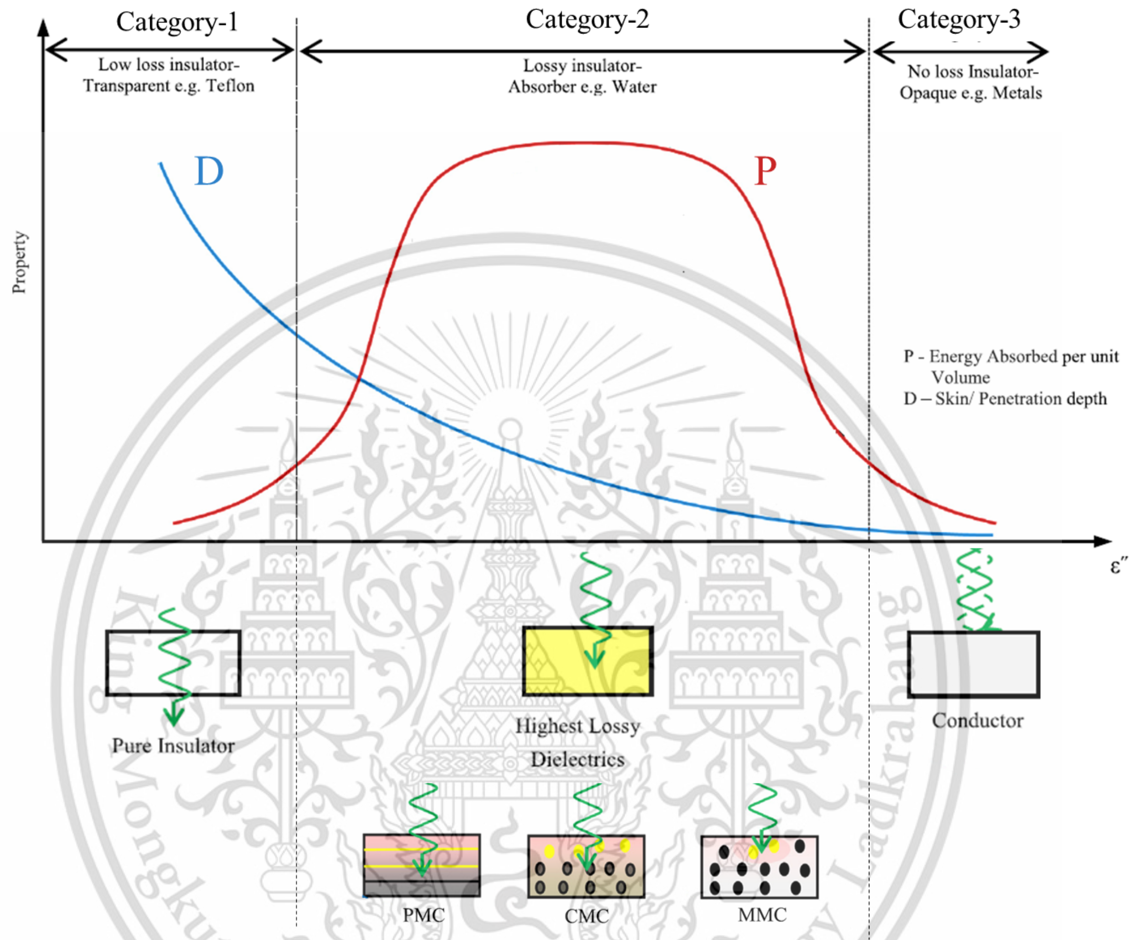


Figure 2.2 The characteristics of microwave absorption and penetration in different types of material. Adapted from [2].

2.3 Microwave heating mechanism

The heating of material using a microwave can occur depending on how the material responds to the electric and magnetic fields of the microwave. Two important parameters which describe the response of material to an electric field are the permittivity or dielectric constant (ε') and dielectric loss (ε''). The permittivity or dielectric constant (ε') of material represents its capability to retain electric energy, whereas dielectric loss (ε'') represents its ability to convert the electric energy to heat. In addition, the reaction of materials to a magnetic field can also be evaluated by permeability (μ') which indicates the ability of material to store magnetic energy and magnetic loss (μ'') which indicated the ability of material to convert magnetic energy to heat.

In general, electric energy is stored in materials by polarization of bound charges. On the other hand, the relaxation of polarized molecules (polarization loss) and the conduction of free electrons (conduction loss) both contribute to the conversion of electric to heat. The dielectric properties of materials can be consider as following:

$$\varepsilon' = \varepsilon'_{\text{dipolar}} + \varepsilon'_{\text{interfacial}} + \varepsilon'_{\text{ionic}} + \varepsilon'_{\text{electronic}} \qquad (2.1)$$

and

$$\varepsilon'' = \underbrace{\varepsilon''_{\text{dipolar}} + \varepsilon''_{\text{interfacial}} + \varepsilon''_{\text{ionic}} + \varepsilon''_{\text{electronic}}}_{\text{Polarization loss}} + \underbrace{\frac{\sigma}{2\pi f}}_{\text{conduction loss}} \qquad (2.2)$$

where σ is the conductivity of material and f is the frequency of the radiation.

The microwave heating of the dielectric insulator materials which have no free charges such as quartz, alumina, and zirconia primary occurs by polarization loss. In contrast, the highly conducting materials such as metals, the heating by conduction loss is the main factor under the electric field of the microwave.

The heating mechanisms for the non-magnetic materials such as water, Al, Cu, polymers, and ceramics, are only occurred by the effect of electric field of microwave. The main heating mechanisms include dipolar loss (polarization loss) and conduction loss. In dielectric insulator materials, heating by dipolar loss occurs predominantly, while for the conductive materials, the heating by conduction loss predominates.

The dielectric properties are often expressed as their relative values with respect to the free space permittivity (ε_0= 8.854 x 10^{-12} Farad/m) as described below.

$$\varepsilon_r' = \frac{\varepsilon'}{\varepsilon_0} \quad \text{and} \quad \varepsilon_r'' = \frac{\varepsilon''}{\varepsilon_0} \tag{2.3}$$

Additionally, it should be noted that the important parameters which indicate the penetration of the microwave are the frequency of the radiation, permittivity/permeability of the material, and loss tangent (tan δ_e). The loss tangent can be defined as follows.

$$\tan \delta_e = \frac{\varepsilon_r''}{\varepsilon_r'} \tag{2.4}$$

2.3.1 Dipolar loss (polarization loss)

In dielectric insulator materials, the dipolar loss is highly significant because dipoles are typically induced when exposed to an external electric field. Examples of dielectric materials include water, food products, ceramics, CMC, and PMC. Figure 2.3 illustrates the process of the molecular dipoles in water (with positive polarity on hydrogen atom and negative polarity on oxygen atom) rotate themselves to stay in phase with the oscillating electric field **E**. The resistances of dipoles motion in the terms of internal force, elastic force, frictional force, and molecular force, cause the increase in molecular kinetic energy and result in volumetric heating. Therefore, the temperature of the material increases rapidly as a result of the increase in kinetic energy of all dipoles in the material.

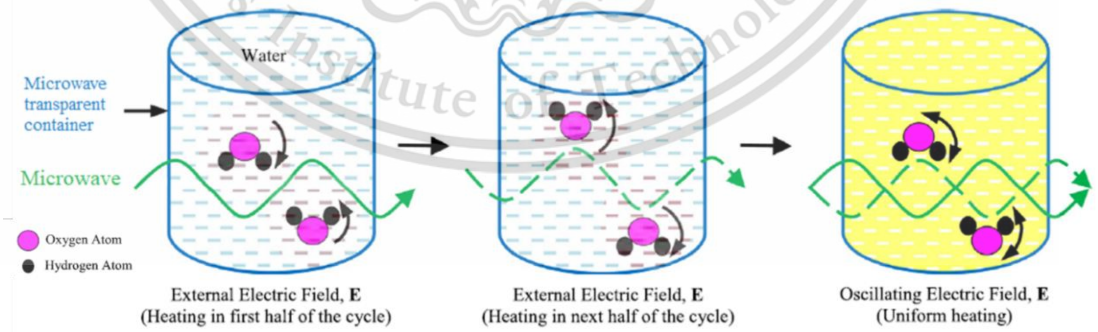
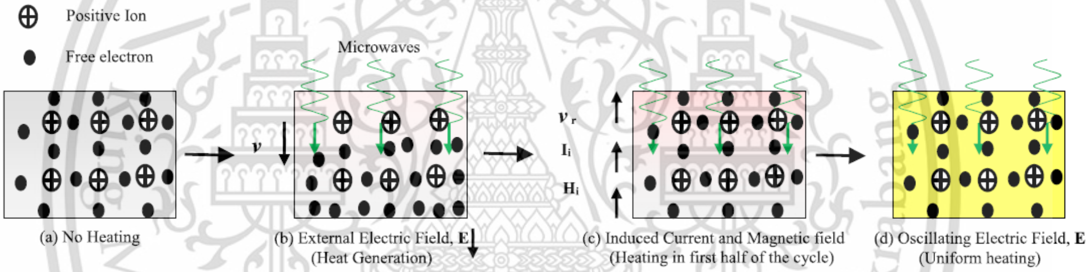


Figure 2.3 Heating mechanism in dipolar loss [2].

2.3.2 Conduction loss

The schematic of conduction mechanism in conductive material is shown in Fig. 2.4. Conduction loss is predominant in microwave processing of pure metals, metal alloys and semiconductors such as Cu, Al, Si, Fe, Ni, and MMC. These materials contain intrinsic free electrons as shown in Fig. 2.4a. When an external electric **E** is applied, the free electrons begin to move with velocity v in the direction of **E** (Fig. 4b). Since these materials have significantly high conductivity, the **E** field is rapidly attenuated inside the materials, resulting in a large current (Ii) induced (Fig. 4c). Consequently, an induced magnetic field (**Hi**) is generated in the reverse direction of external magnetic field inside the material. The induced magnetic field produces a force on travelling electrons, causing conducting electrons to move in the opposite direction at velocity v_r. Therefore, kinetic energy in material is increased from the restriction of electron mobility caused by inertial forces, elastic forces, frictional forces, and molecular interaction forces. The oscillating electric field rapidly repeats this process rapidly, resulting in volumetric and uniform heating within the material as shown in Fig. 4d.



(a) No Heating
(b) External Electric Field, **E** (Heat Generation)
(c) Induced Current and Magnetic field (Heating in first half of the cycle)
(d) Oscillating Electric Field, **E** (Uniform heating)

Figure 2.4 Heating mechanism in conduction loss [2].

2.4 Comparison of conventional heating and microwave heating

The mechanism of microwave heating is completely different from that of traditional heating. Microwave heating produced a reverse temperature gradient when compared to conventional heating as shown in Figure 2.5.

2.4.1 Conventional heating

Conventional heating is the process that heat is transferred from one place to another due to the movement of fluid which is known as thermal convection. Heat is generated from the heating source then transfer by surrounding air to the surface of sample. The heat is gradually transferred from surface to the core of sample by conduction causing the temperature gradient in which outer surface is hotter than its interior. Therefore, heating with the conventional method takes a lot of time and energy.

2.4.2 Microwave heating

The material that can absorb microwave generally has a dipole molecule when that dipole molecule receives an external electric field such as microwave it will reorient itself in order to be in the same phase with that electric field so that internal elastic frictional and molecular interaction forces resist their changes which increase kinetic energy and result in “volumetric heat”. This process occurs rapidly and requires lower energy compared to conventional heating. However, this process also confronts the problem that the temperature of the core and surface are different because at the surface of the sample heat will transfer to the atmosphere by the conventional model.

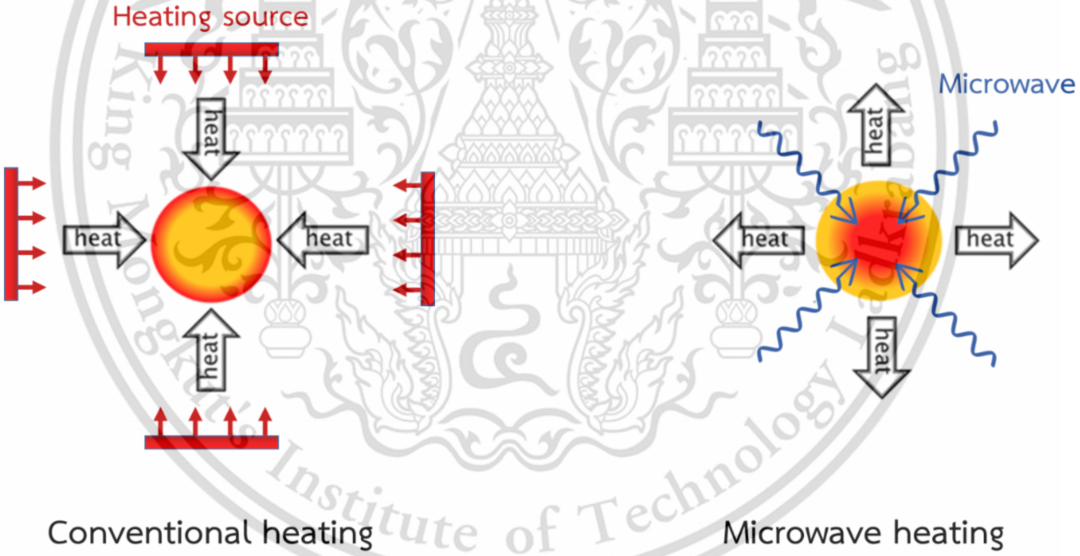


Figure 2.5 Patterns of heat generation by conventional and microwave heating.

2.4.3 Microwave hybrid heating (MHH)

The process of microwave heating using heat generated by the susceptor is commonly known as microwave hybrid heating as shown in Figure 2.6. To reduce the different temperature between the core and surface of sample, susceptor can be used to improve their heating process. Microwave heating with a susceptor is a sample method that sample is heated by both microwave heating and conventional heating. Additionally, microwave hybrid heating can be applied to heat the transparent materials which cannot absorb microwave by themselves.

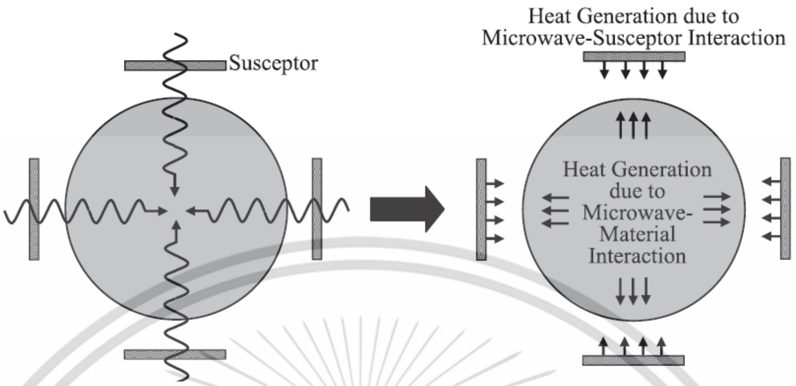


Figure 2.6 Microwave hybrid heating (MHH) [4].

2.5 Microwave susceptor

2.5.1 Susceptor properties

The susceptor is a highly loss material such as SiC and carbon, that can absorb microwave and convert to heat efficiently. As a result, it can rapidly be heated by microwave. The addition of susceptor to the microwave heating process allows it to be heated by microwave and then transfer heat to sample in the conventional manner. The heat generated by susceptor compensates for heat that sample loss to the atmosphere so the temperature of surface and core will equal. Table 2.1 shows a comparison of penetration depth (d_p) and loss tangent $(\tan\delta_e)$ values of reflector, microwave transparent, and microwave absorbing materials.

Reflector materials have extremely low penetration depth in the order of μm, implying that microwave is reflected at the surface of reflector material. Since they have no $\tan\delta_e$ and cannot be heated by microwave, this type of material cannot be used as susceptor. For microwave transparent materials, they have high penetration depth and low $\tan\delta_e$, so microwaves pass through them without any absorption. On the other hand, microwave absorbing materials have a high value of $\tan\delta_e$ and their penetration depth are in the order of cm. So, this type of material can absorb and convert microwave into heat, and they have an excellent potential to be susceptor.

The bulk materials that are good microwave absorbers include SiC and several types of carbon such as carbon black, graphite, and charcoal.

Table 2.1 Comparison of penetration depth (d_p) and loss tangent ($\tan\boldsymbol{\delta_e}$) values of reflector, microwave transparent, and microwave absorbing materials [4].

Reflector		Microwave transparent			Microwave absorber		
Bulk metal	d_p (μm)	Material	$\tan\delta_e$	d_p (m)	Material	$\tan\delta_e$	d_p (cm)
Al[a]	1.7	Alumina[a]	0.001	12.65	SiC[a]	0.37	1.93
Cu[a]	1.3	Fused quartz[a]	0.0003	75.73	Water[a]	0.15	3
Au[a]	1.5	Borosilicate glass[a]	0.0012	15.7	Carbon Black[d] (20 μm)	0.23	5.75
Ag[a]	1.3	Teflon[a]	0.00048	56.4	Graphite Powder[d] (20–80 μm)	0.36–0.67	1.34–2.09
Zr[a]	6.7	Mullite[a]	0.0015	10.2	Graphitized Carbon Powder (60–80 mesh)[e]	0.4–1	0.5–0.9
		Porous alumina fiberboard[a]	3×10^{-5}	421	Activated Carbon[f]	0.31–0.9	0.7–3.43
		Yttria stabilized zirconia[b]	0.0011	>5	Charcoal[g]	0.14–0.38	6–11
		PVC[a]	0.0056	4.03	Carbon Fibers[h]	0.45–0.5	0.5–0.7
		Polystyrene[a]	0.0003	76.2	Carbon Nanotube[i]	1.11	0.2
		Silicon[a]	<0.012	>3.96			
		Silicon Nitride[b]	<0.001	>12.2			
		Boron Nitride[c]	<0.0005	>35			

2.5.2 Design of susceptor

In microwave hybrid heating, the effect of heating depends on the design of the susceptor. Susceptors should provide the pre-heating to the material, but they should not totally block material from the microwave. A design of the susceptor configuration and arrangement such as the thickness, the amount, and the position of the susceptor should be considered. Figure 2.7 shows the sample of the arrangement of susceptor used in microwave hybrid heating.

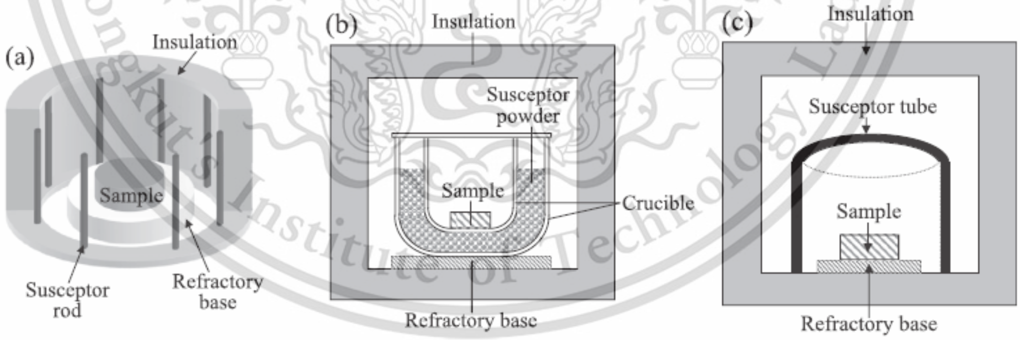


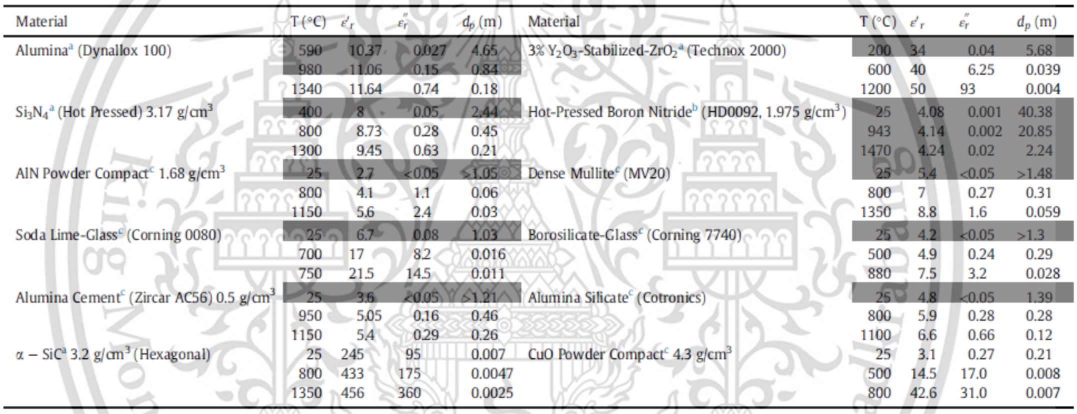
Figure 2.7 Microwave hybrid heating configuration (a) rod-like susceptor, (b) powder susceptor, and (c) tube susceptor [4].

2.6 Sintering of ceramics by microwave

In conventional heating process of ceramics, heating begins at the surface of sample, and the core of the sample often has a lower temperature than the surface. This phenomenon may result in a distinct microstructure between the surface and core of ceramic and thus affect the specimen quality. The non-uniform temperature during heating also makes a product at risk of cracking, particularly for ceramic materials that need to sinter at high temperature, such as alumina and silicon nitride. Furthermore, the long dwell time at high temperature causes samples to often encounter the grain growth problem.

Microwave sintering can offer a solution to these problems. Microwave heating not only reduces the dwell time for sintering but also provides the homogeneous heating process. Table 2.2 shows the dielectric properties of ceramic materials at different temperatures. Generally, most ceramics have low lossy at room temperature, but the dielectric properties increase with temperature. Therefore, susceptor addition is the majority role in sintering ceramic by microwave.

Table 2.2 The dielectric properties of ceramic materials at different temperature [4].



Material	T (°C)	ε'_r	ε''_r	d_p (m)	Material	T (°C)	ε'_r	ε''_r	d_p (m)
Alumina[a] (Dynallox 100)	590	10.37	0.027	4.65	3% Y_2O_3-Stabilized-ZrO_2[a] (Technox 2000)	200	34	0.04	5.68
	980	11.06	0.15	0.84		600	40	6.25	0.039
	1340	11.64	0.74	0.18		1200	50	93	0.004
Si_3N_4[a] (Hot Pressed) 3.17 g/cm^3	400	8	0.05	2.44	Hot-Pressed Boron Nitride[b] (HD0092, 1.975 g/cm^3)	25	4.08	0.001	40.38
	800	8.73	0.28	0.45		943	4.14	0.002	20.85
	1300	9.45	0.63	0.21		1470	4.24	0.02	2.24
AlN Powder Compact[c] 1.68 g/cm^3	25	2.7	<0.05	>1.05	Dense Mullite[c] (MV20)	25	5.4	<0.05	>1.48
	800	4.1	1.1	0.06		800	7	0.27	0.31
	1150	5.6	2.4	0.03		1350	8.8	1.6	0.059
Soda Lime-Glass[c] (Corning 0080)	25	6.7	0.08	1.03	Borosilicate-Glass[c] (Corning 7740)	25	4.2	<0.05	>1.3
	700	17	8.2	0.016		500	4.9	0.24	0.29
	750	21.5	14.5	0.011		880	7.5	3.2	0.028
Alumina Cement[c] (Zircar AC56) 0.5 g/cm^3	25	3.6	<0.05	>1.21	Alumina Silicate[c] (Cotronics)	25	4.8	<0.05	1.39
	950	5.05	0.16	0.46		800	5.9	0.28	0.28
	1150	5.4	0.29	0.26		1100	6.6	0.66	0.12
α – SiC[a] 3.2 g/cm^3 (Hexagonal)	25	245	95	0.007	CuO Powder Compact[c] 4.3 g/cm^3	25	3.1	0.27	0.21
	800	433	175	0.0047		500	14.5	17.0	0.008
	1350	456	360	0.0025		800	42.6	31.0	0.007

2.6.1 Alumina and zirconia

Alumina and zirconia are microwave transparent ceramic that have been successfully sintered by microwave hybrid heating (MHH) with SiC rods as a susceptor. The defect free alumina and zirconia samples were sintered by MHH at about 100-200°C lower temperature than conventional sintering [5,6]. The properties of the final product from microwave sintering and conventional sintering are different depending on temperature, dwelling time, and the heating rate. For example, De et al. [7] reported that high purity alumina was effectively sintered by microwave hybrid heating at 1500°C for 30 min, and the resulting alumina had a uniform structure with density greater than 95%TD. However, the density of alumina sample sintered by conventional process did not reach 95%TD even with a longer sintering time for 60 min at 1500°C. Zhao et al.

[8] reported that sintering of yttria stabilized zirconia was successful done using tubular SiC as a susceptor in microwave hybrid heating. The microwave hybrid sintering also provided a shorter time of sintering and more efficient energy consumption as shown in Figure 2.8.

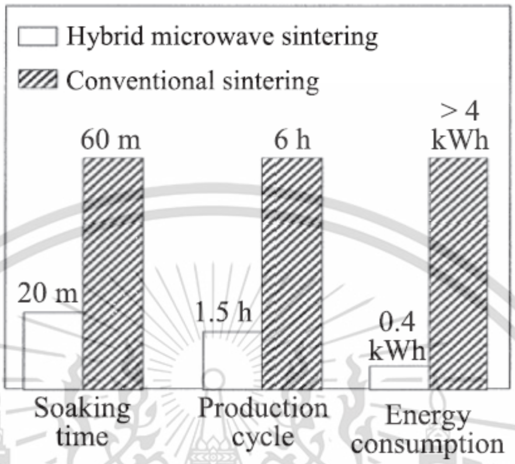


Figure 2.8 Comparison of microwave hybrid heating and conventional heating for sintering yttria stabilized zirconia [8].

2.6.2 Mullite and porcelain

Mullite and porcelain are not good at absorbing microwaves like alumina and zirconia at room temperature. Many researchers reported the use of susceptor for assisting the sintering of mullite and porcelain. Souto et al. [9] reported that mullite powder was successfully sintered by microwave hybrid heating using SiC as a susceptor. When compared to conventional sintering, MHH sintering took less than 20% of the processing time. Satapathy [10] reported that porcelain rods (12 mm in diameter and 150 mm in length) were successfully sintered at 1240 °C for 30 min by using SiC as susceptor. The MHH sintering produced non-crack and uniform sintered products, and the result was equivalent to the conventional sintering for 6 h. Figure 2.9 shows the sintering profiles of porcelain-based ceramics (dental ware and sanitary ware) by microwave hybrid heating and conventional heating [10].

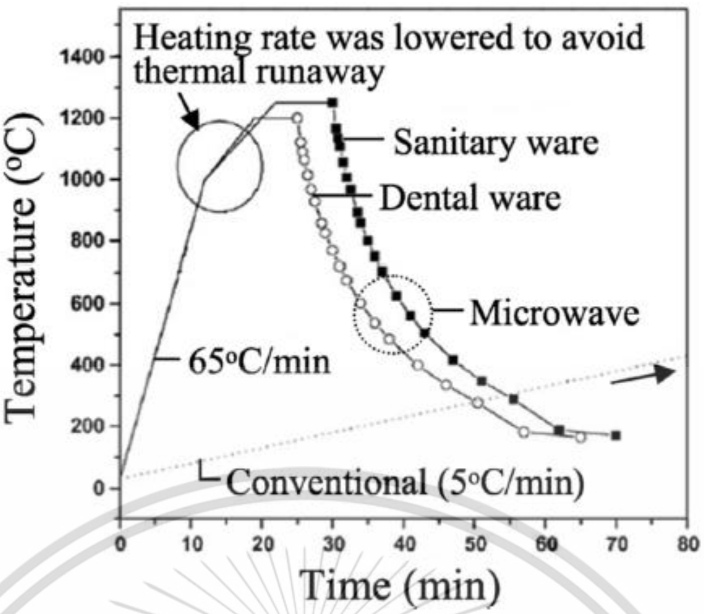


Figure 2.9 Sintering profiles of porcelain-based ceramics (dental ware and sanitary ware) by microwave hybrid heating and conventional heating [10].

2.7 Literature review

2.7.1 The effects of microwave sintering compared with conventional heating

According to the review from Oghbaei and Mirzaee [11], microwave sintering has many advantages over conventional sintering. Microwave sintering consumes less energy lower than conventional heating due to the shorter sintering time. Microwave sintering improves density of sample and provides uniform grain size distribution. Also, microwave sintering can deliver excellent physical and mechanical properties.

Madhan and Prabhakaran [12] reported the comparative study of Al_2O_3-SiC ceramic composites prepared by conventional and microwave sintering at 1500°C. The 10wt% SiC samples sample that sintered by microwave offered higher Vickers hardness compared to a conventional one (24.6 and 22.6 GPa, respectively). The uniform grain formations also obtained from microwave sintering.

Guillen et al. [13] reported the sintering of 3Y-TZP/TiO_2 materials at 1500°C by microwave and conventional sintering. The samples prepared by microwave sintering showed high density with grain size and hardness similar to those obtained by conventional sintering. In addition, microwave sintering effectively reduces power consumption because microwave heating cycle was only 40 min, whereas the conventional process was 2 and 6 h. Microwave sintering can save total energy input of 80%.

Lyra et al. [14] prepared lightweight aggregate red clay with addition of sugarcane bagasse ash and sintered using microwave oven. In comparison to the lightweight aggregate sintered in electric oven for 1 h, aggregates sintered in a microwave oven only for 10–20 min showed higher compressive strength, lower in water absorption and refinement in the microstructure.

Zeng et al. [15] reported a comparative study between the microwave and conventional sintering of Li_2TiO_3–Li_4SiO_4 biphasic ceramic pebbles. The biphasic ceramic pebbles obtained by microwave sintering had a greater microstructure and mechanical properties compared to conventional sintering. Microwave sintering produced microstructure samples with uniformly distributed grain size, higher density, and crush load than conventional sintering.

Table 2.3 Summary of the effects of microwave sintering compared with conventional heating

Authors	Topics	Main conclusion
Oghbaei and Mirzaee (2010) [11]	Microwave sintering application compared to conventional sintering	Microwave sintering consumes less energy lower due to shorter sintering time.
Madhan and Prabhakaran (2018) [12]	Microwave versus conventional sintering: Microstructure and mechanical properties of Al_2O_3-SiC composites	Al_2O_3–SiC composites prepared by microwave sintering offer higher Vickers hardness and more uniform grain formations.
Guillen et al. (2021) [13]	Dielectric, mechanical, and thermal properties of ZrO_2–TiO_2 materials obtained by microwave sintering at low temperature	The sinterability of 3Y-TZP/TiO_2 materials is strongly enhanced using microwave sintering. The sintering time decreases form 2-6 h (CV) to 15 min (MW).
Lyra et al. (2019) [14]	Reuse of sugarcane bagasse ash to produce a lightweight aggregate using microwave oven sintering	The microwave reduced time and energy consumption in the production of aggregates. The sintered aggregates in a microwave oven showed higher compressive strength.
Zeng et al. (2019) [15]	Fast fabrication of high quality Li_2TiO_3–Li_4SiO_4 biphasic ceramic pebbles by microwave sintering: In comparison with conventional sintering	Microwave sintering produces ceramic pebbles with significantly improved microstructure and mechanical properties over conventionally sintered pebbles.

2.7.2 The effects of microwave sintering with a susceptor

According to the review of Yahaya et al. [1], the use of a susceptor has an advantage over using only a microwave as an energy source. Susceptors act as a thermal generating source, improve the heating uniformity, reduce temperature gradient, and solve thermal runaway problems. Microwave hybrid heating (MHH) using susceptor also reduces processing time and saves energy. Microwave hybrid heating is also an effective way to process materials that have low microwave absorption such as transparent and reflecting materials. The most common susceptor materials are silicon carbide (SiC), carbon (C), and magnetite (Fe_3O_4).

Bhattacharya et al. [4] presented a review on the susceptor assisted microwave processing of materials and reported that susceptor enhance microwave processing because it provides two-ways heating (microwave and conventional heating along together). The susceptor improves the uniformity of microwave heating and reduces the heat loss of sample. The heating rate of MHH is also higher than that of only direct microwave heating or conventional heating and it reduces the risk of crack formation.

Bhoi et al. [16] studied the type of susceptor materials (graphite, charcoal, and silicon carbide) for microwave hybrid heating. In a microwave with 900 W and 2.45 GHz, the graphite boat was found to give a maximum temperature of 350°C at 70 min, while charcoal and silicon carbide reached 410°C and 255°C at only 10 min. However, it was reported that silicon carbide susceptor are suitable to use in high-temperature applications due to it is stable at high temperature.

Peroglio et al. [17] studied high-speed microwave sintering of robocast porcelain using a multimode microwave (2.45 GHz, 3 kW). In the microwave sintering process without susceptor heating, porcelain required an energy of 1450 W and had a duration of a process cycle of 86 min. In contrast, microwave sintering with SiC susceptor reduced energy used to 900 W and process cycle duration was only 56 min due to the effect of high power absorption of silicon carbide.

Huang et al. [18] studied the sintering of Al_2O_3 ceramic by hybrid microwave sintering (HMS). The thermal residual stress distribution was used to evaluate the sinterability of sample. It was found that the sample from HMS was homogenously sintered with an improvement in mechanical properties compared to the one from the conventional sintering. The microwave sintering with SiC susceptor provides two directional sintering because microwave heating direction was from inside to outside, whereas susceptor heating direction was from outside to inside. So, the uniformed heat can improve mechanical properties of sample.

Table 2.4 Summary of the effects of microwave sintering with a susceptor.

Authors	Topics	Main conclusion
Yahaya et al. (2014) [1]	Microwave Hybrid Heating of Materials Using Susceptors- A brief review	The use of a susceptor has an advantage over using only a microwave as an energy source. MHH reduces processing time and saves energy and can process materials that have low microwave absorption like transparent and reflecting materials.
Bhattacharya et al. (2016) [4]	A review on the susceptor assisted microwave processing of materials	The rapid initial heating via susceptor executes the energy efficient microwave processing for the poorly microwave absorbing materials.
Bhoi et al. (2019) [16]	A study on microwave susceptor material for hybrid heating	Silicon carbide have a high heating rate and stable at high temperature.
Peroglio et al. (2022) [17]	A parametric study of conventional and high-speed microwave sintering of robocast porcelain	Adding a susceptor to microwave sintering can reduce sintering time and energy use in the sintering process.
Huang et al. (2008) [18]	Improving sinterability of ceramics using hybrid microwave heating	The use of SiC susceptor provide homogeneous heating of samples which improve their mechanical properties.

Chapter 3

Research methodology

The experiment was divided into two parts. The first part is the experiment to find the most effective way to use susceptor in microwave hybrid heating. The second part is comparison of sintering of clay pellets by microwave hybrid sintering (MHS) and conventional sintering (CVS). The physical and mechanical properties of clay pellets sintered from MHS and CVS were systematically investigated.

3.1 Materials and equipment

3.1.1 Materials

In this research, clay pellets were made in the laboratory with a mix of 3 ingredients including red clay powder (Pantong soil from Chonburi province, Thailand), expanded perlite (Klongyang Co., Ltd., Thailand), Carboxymethylcellulose or CMC (Chemipan Corp., Thailand) The raw materials are shown in Figure 3.1.

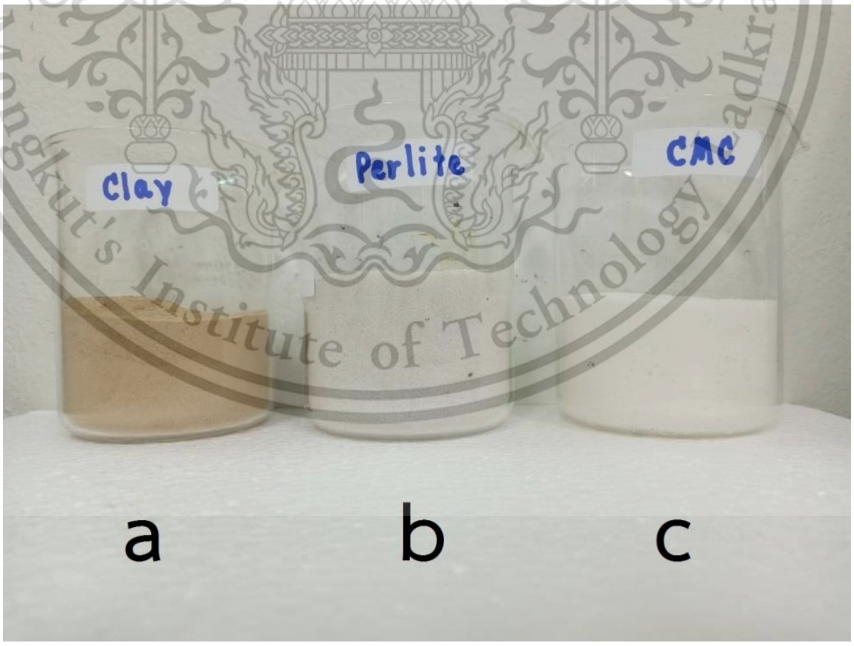
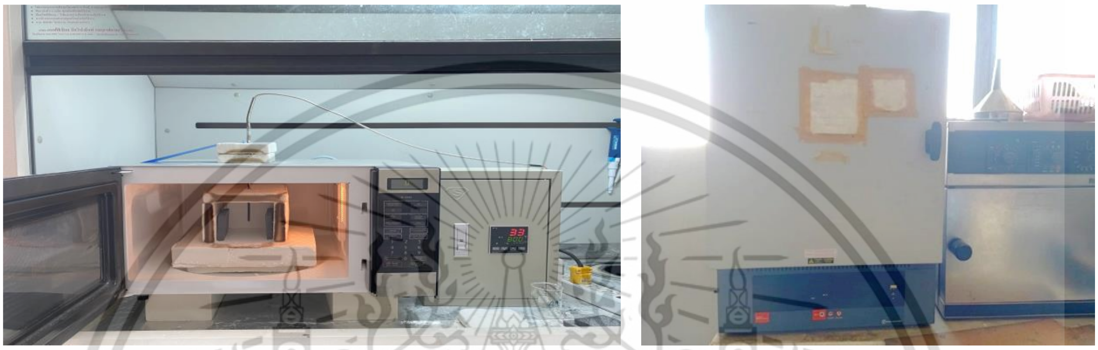


Figure 3.1 Raw materials: (a) red clay powder, (b) expanded perlite, and (c) CMC.

3.2.2 Equipment

To sinter clay pellets in microwave hybrid heating system, microwave oven (Sharp, model R-3901, power output of 1200 W) was used to modified. The conventional sintering was carried out by laboratory electric furnace (Isotemp® Muffle Furnace 550 Series, Fisher Scientific). In measure the temperature in a microwave oven thermocouple type k was used as shown in Figure 3.2.



(a) (b)

Figure 3.2 Sintering equipment: (a) modified microwave oven and (b) electric furnace.

3.3 Experimental procedure

3.3.1 Microwave hybrid heating system testing

The SiC plate used as susceptor in this experiment has a thickness of 1.2 cm. The SiC plate was cut to 2 dimensions. The dimension of SiC that was placed at left- and right-side of the sample was 8.5 cm x 6.0 cm in height x width, as shown in Figure 3.3. The dimension of SiC that was placed at the bottom of the sample was 10 cm x 10 cm in height x width. The experiment to determine the efficiency of heating by microwave oven was designed in three different configurations, each with a different number of SiC plates as shown in Figure 3.4. Each SiC configuration was placed in the insulation box made from Alumina-Silica ceramic fiber board insulator (ISOLITE) with a thickness of 5 cm.

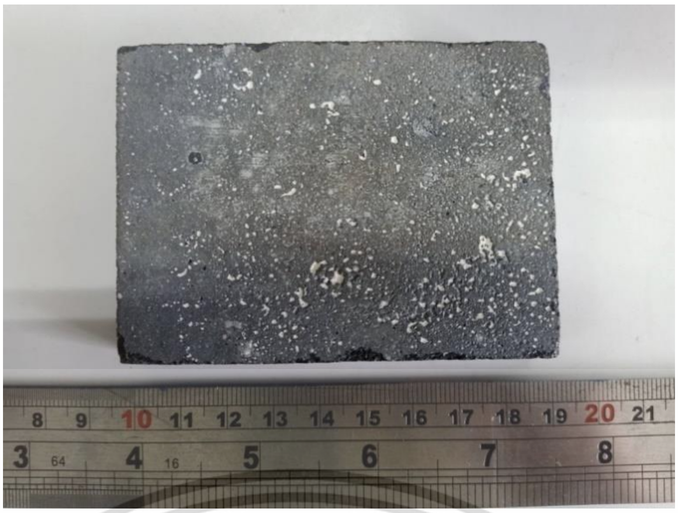
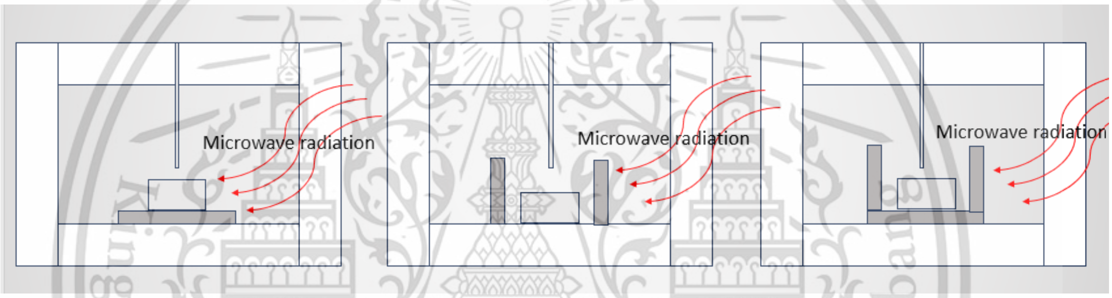


Figure 3.3 SiC susceptor plate.



(a) (b) (c)

Figure 3.4 SiC configuration; (a) 1 SiC plate, (b) 2 SiC plates, and (c) 3 SiC plates.

3.3.2 Microwave hybrid heating conditions

One of the main points of this research is to save energy by using microwave sintering because microwave heating generally uses less energy than conventional heating. In addition, we investigated various microwave power levels in order to determine the most effective power level for sintering. In this experiment, microwave power at 20, 40, 60, 80 and 100% (240, 480, 720, 960, and 1200 watts) were used. The temperature in the microwave was recorded every 1 min. Then the temperature in microwave chamber was measured in relation to time. The test was scheduled for 30 minutes or until the temperature reached 900°C.

3.3.3 Preparation of green clay pellets

The green clay pellets were prepared for a sintering experiment. First, red clay powder was mixed with expanded perlite at a weight ratio of 7:3. The 0.05 wt% CMC solution was used as a binder. The 375 ml binder was added to a 500 g batch of perlite mixed-clay and then mixed by laboratory mixer. The clay pellets were shaped to a diameter of about 10.5-11 mm (1.2 g each) by molding pill machine. After that, the clay pellets were dried by electric oven at 100°C for 24 h.

3.3.4 Sintering of clay pellets

After drying, 20 pellets of clay pellets were put into an alumina crucible and heated by microwave hybrid sintering system with SiC susceptors as shown in Figure 3.5. The experiment was conducted at 5 different temperatures i.e., 700, 750, 800, 850, and 900°C. The clay pellets were also sintered by conventional furnace at 700, 750, 800, 850, and 900°C to compare with MHS samples. Figure 3.6 illustrates the comparison of temperature profiles for Microwave Hybrid Sintering (MHS) and Conventional Sintering (CVS) to reach the maximum temperature of 900°C. Table 3.1 presents a summary of the energy consumption during sintering, comparing microwave hybrid heating and conventional heating.

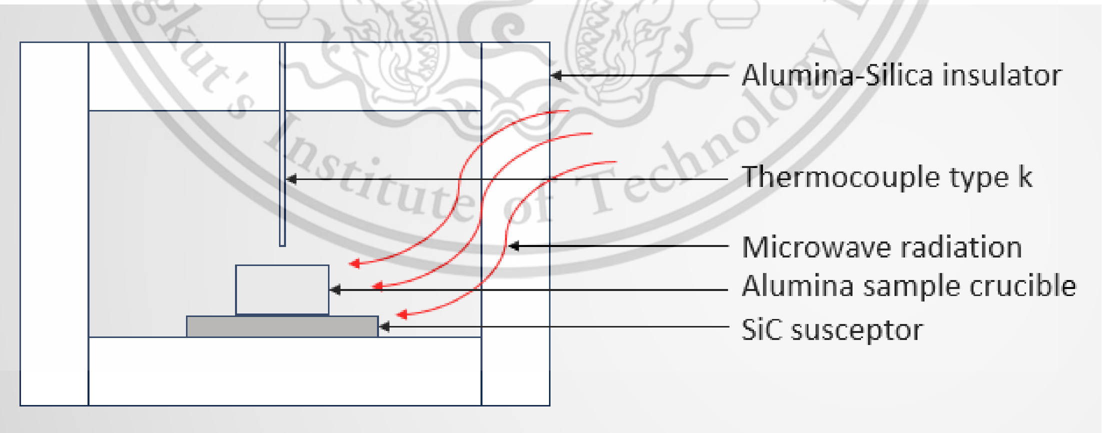


Figure 3.5 The setup of microwave hybrid sintering (MHS) for clay pellets.

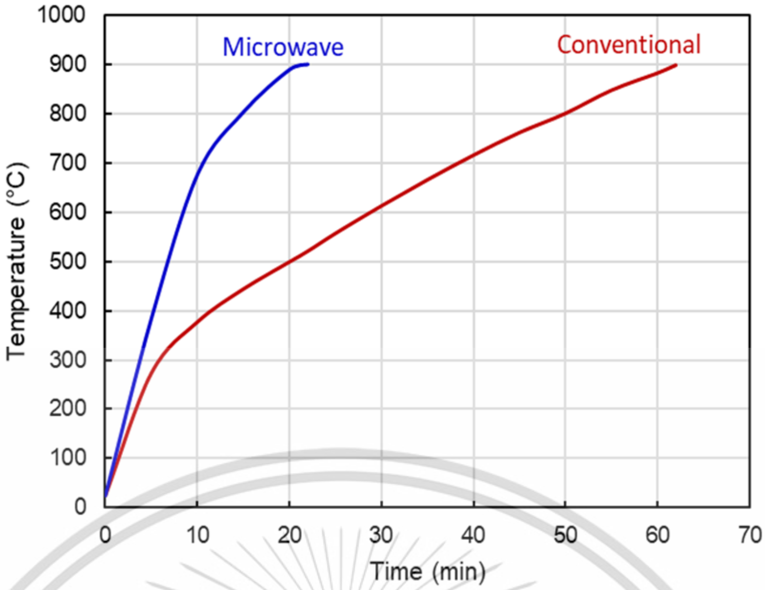


Figure 3.6 Comparison of temperature profile for MHS and CVS to reach the maximum temperature at 900°C.

Table 3.1 Summary of the energy consumption during sintering, comparing microwave hybrid heating and conventional heating

Temperature (°C)	Power consumption (W-h)	
	Conventional	Microwave
700	2272	266
750	2921	290
800	3245	339
850	3634	411
900	4024	532

3.4 Characterizations

In this study, the characterization of clay pellets are physical properties, phase composition, and mechanical properties. The details of the characterization method are as follows.

3.4.1 Physical properties

The investigation of bulk density, porosity, and water absorption were carried out according to the ASTM C20 and ASTM C1185 [16,17]. The clay pellets were put into and oven at 100 °C for 24 h then left cooling. Next, clay pellets were put into boiling water at 100 °C for 2 h then left cooling at atmosphere temperature and soaked for 24 h and then weighed in water (S). The clay pellets were then blotted with a damp cloth before being weighed in the air; this weight is known as saturated weight (W). The volume of specimens (V) was calculated from $V = W - S$. The following are the bulk density, porosity, and water absorption of clay pellets.

$$\text{Bulk Density} = \frac{W}{D} \tag{3.1}$$

$$\text{Porosity} = \frac{W-D}{V} \times 100 \tag{3.2}$$

$$\text{Water absorption} = \frac{W-D}{D} \times 100 \tag{3.2}$$

3.4.2 Phase composition analysis

The characterization of phase composition of clay pellets was performed by using X-ray diffraction (XRD) (Miniflex 600, Rigaku, Japan) as shown in Figure.3.7.

Figure 3.7 X-ray diffractometer (Miniflex 600, Rigaku, Japan).

3.4.3 Mechanical testing

The uniaxial compression test was employed to determine the crushing strength (σ_c) of the pellets. Figure 3.8 shows the Universal testing machine (Bravo TG-64) for crushing strength test. The pellet was placed between 2 parallel rigid plates and the load speed was 2 mm/min until a sample cracked. The load that makes the pellet crack was recorded as F_c and r is the radius of sample. The stress of sample was defined using the following equation.

$$\sigma_c = \frac{F_c}{\pi r^2} \tag{3.4}$$

Figure 3.8 Universal testing machine for crushing strength test.

Chapter 4

Results and discussion

This chapter presents the results of an experiment and subsequent discussion. It is divided into two main sections. The first section examines the effect of the number of SiC plates and microwave power on the temperature of the microwave hybrid heating system. The second section investigates phase compositions, physical and mechanical properties, as well as the microstructure of clay pellets. This analysis involves a comparison between specimens sintered via microwave heating and those subjected to conventional heating methods.

4.1 The effects of SiC layout and microwave power on temperature profile

The study examined the correlation between the quantity of SiC susceptor plates, the power of the microwave oven, and the resulting temperature inside the heating chamber. Three distinct configurations, each featuring a different number of SiC plates ranging from 1 to 3 plates, were investigated. The layout of the susceptor is depicted in Figure 3.4 in the previous chapter, while microwave power was varied in a range from 20% to 100% (equivalent to 240 to 1200 watts).

According to the experiment results, the temperature inside the microwave oven increased over time and rose in response to an increase in electrical power. Figure 4.1 illustrates the temperature profiles within the microwave oven when employing a single SiC susceptor plate. After 30 minutes, utilizing a single SiC susceptor and 20% electrical power, the highest recorded temperature was 334°C. Subsequently, as the electrical power increased to 40% and 60%, the highest temperatures increased to 640°C and 689°C, respectively. Similarly, the temperature increased with an elevation in electrical power to 80% and 100%, reaching maximum temperatures of 870°C and 889°C, respectively.

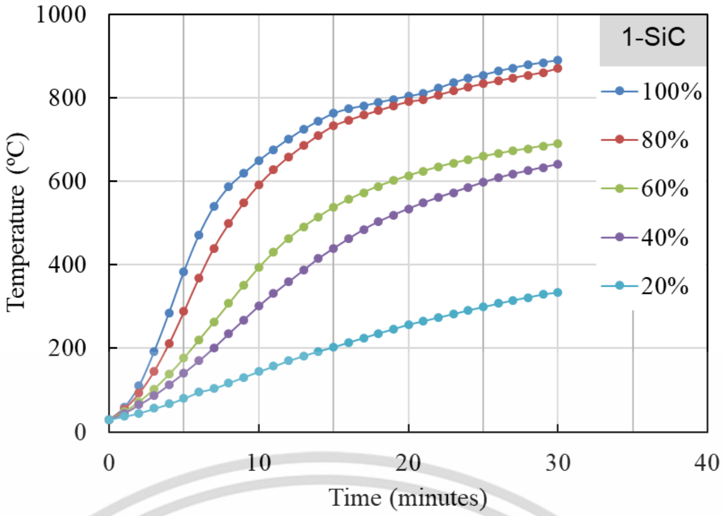


Figure 4.1 Temperature profiles during microwave hybrid heating using a single SiC susceptor plate.

Figure 4.2 depicts the temperature profile when employing two SiC susceptor plates. The highest temperature achievable using 20% electrical power for 30 minutes was limited to 226°C. Subsequently, the maximum temperature increased to 650°C with 40% electrical power. With further increase in electrical power to 60%, 80%, and 100%, the temperature rose rapidly, reaching 900°C before the 30 min. Notably, at 60% electrical power, the internal temperature reached 912°C after 26 min, and at 80% electrical power, it reached 915°C after 17 min. Ultimately, elevating the electrical power to 100% resulted in the maximum temperature of 925°C in only 15 min.

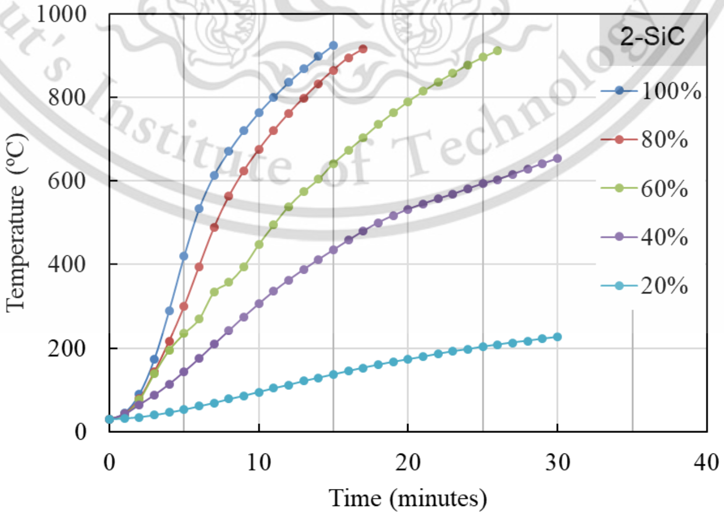


Figure 4.2 Temperature profiles during microwave hybrid heating using two SiC susceptor plates.

In Figure 4.3, the temperature profiles of the microwave oven with three SiC susceptor plates are depicted. The highest temperature recorded was 314°C when applying 20% electrical power. Subsequently, with increases in electrical power to 40% and 60%, maximum temperatures of 591°C and 888°C were achieved, respectively. The peak temperature was reached at 80% electrical power, reaching 888°C. Finally, after 30 minutes of operation at 100% electrical power, the maximum temperature of the oven reached 913°C.

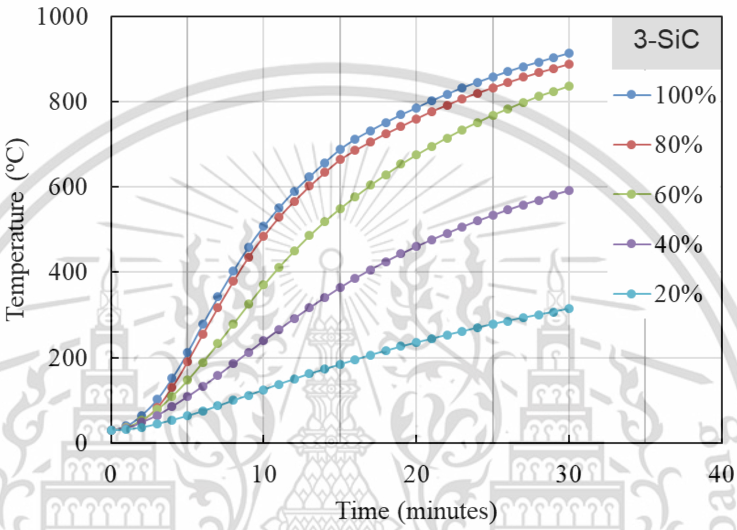


Figure 4.3 Temperature profiles during microwave hybrid heating using three SiC susceptor plates.

The study revealed that increasing the number of SiC susceptor plates from two to three did not enhance the heating rate; instead, it led to a decrease in efficiency. This decline in efficiency can be attributed to wave interference and reduced microwave energy absorption resulting from an excess of susceptors. The presence of an excessive number of susceptors may hinder the propagation of microwaves, inducing standing waves and a reduction in penetration depth.

In microwave heating, standing waves can manifest when waves reflected from different surfaces within the oven interfere with each other. When microwaves encounter a reflective surface like metal or a SiC susceptor, they may be reflected towards the source. If these reflected waves combine with incoming waves, they can create fixed areas of high and low intensity, forming standing wave patterns in space. As a result, the findings of this study highlight the importance of carefully optimizing the number and placement of susceptors to achieve maximum heating efficiency in microwave hybrid heating.

This study investigated the optimal heating conditions for microwave hybrid heating (MHH), concluding that the use of two silicon carbide susceptor plates yielded the most efficient heating. When applying microwave power at 100% (1200 W), the chamber temperature reached 925°C within 15 min. This resulted in a heating rate averaging 62°C/min, which proved notably higher compared to other conditions. Following the determination of the optimal susceptor configuration, this setup was subsequently utilized in an extended study focusing on the sintering of clay pellets.

4.2 Comparative analysis of clay pellets sintered by microwave hybrid sintering (MHS) and conventional sintering (CVS)

In this section, the chosen susceptor configuration, involving two silicon carbide susceptor plates and microwave power set at 100% (1200 W), was applied to investigate its effectiveness in the sintering process of clay pellets. The aim was to explore how this specific susceptor arrangement influenced the sintering outcomes, including phase compositions, physical and mechanical properties, the microstructure, and mechanical strength of the resulting clay pellets. This approach allowed for a more in-depth understanding of the practical applications in the context of sintering materials like clay pellets using microwave hybrid sintering (MHS). A comprehensive comparison with conventional heating methods was conducted. This comparative analysis aimed to evaluate the efficiency and outcomes of the MHS process in comparison to conventional sintering (CVS) methods commonly used in clay pellet fabrication.

4.2.1 Phase composition analysis by XRD

Initially, the phase composition of the main raw materials, Phanthong clay and expanded perlite, was examined using XRD analysis. Figure 4.4 illustrates the XRD pattern of expanded perlite. The pattern reveals a distinct broad peak in the 2-theta range of approximately 20-30 degrees. The broad peak in this region is characteristic of the amorphous structure of silica (SiO_2) since perlite is typically found in nature as volcanic glass in an amorphous form. Additionally, peaks corresponding to Albite

($NaAlSi_3O_8$) were observed, suggesting a minor presence of sodium aluminum silicate mineral with a crystalline structure. Figure 4.5 illustrates the XRD pattern for Phanthong clay. Analysis of the XRD pattern revealed that silica (SiO_2) is the predominant compound, indicated by the highest intensity peak. Minor peaks were also detected, corresponding to halloysite ($Al2Si_2O_5(OH)_4{\cdot}2H_2O$) and brookite ($TiO_2$).

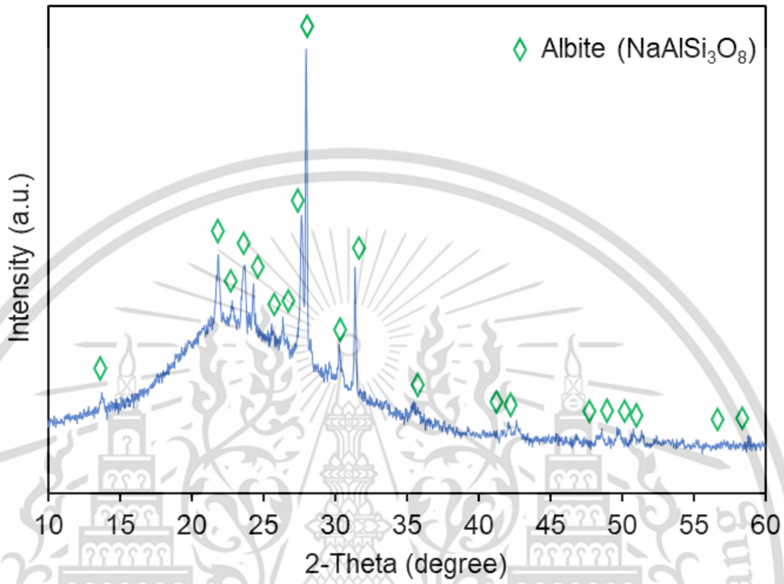


Figure 4.4 XRD pattern of expanded perlite.

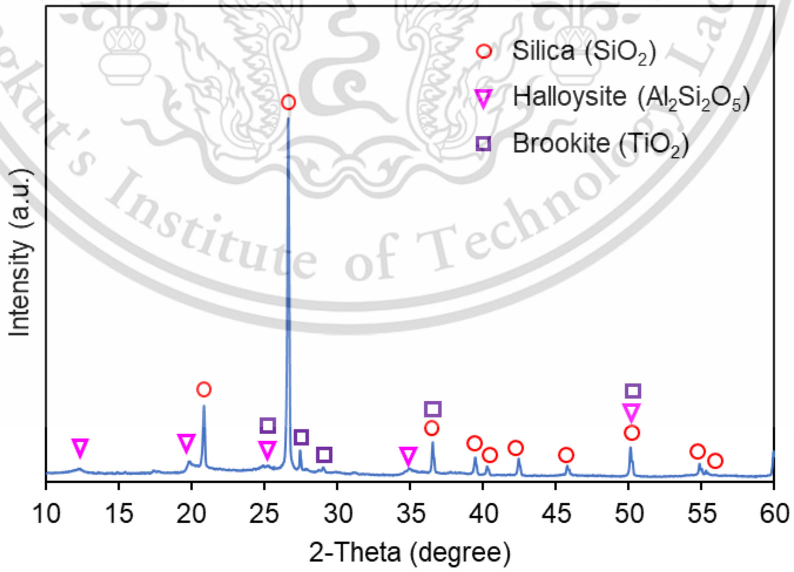


Figure 4.5 XRD pattern of Phanthong clay.

After the sintering process, the phase composition of the porous ceramic pellets was analyzed using XRD. The XRD patterns of pellets produced by microwave hybrid sintering (Figure 4.6) and conventional sintering (Figure 4.7) exhibited similar patterns. The results indicate the predominant presence of silica (SiO_2) as the main phase, accompanied by minor peaks corresponding to halloysite ($Al_2Si_2O_5$) and albite ($NaAlSi_3O_8$) in all samples. The phases present in the sintered pellets are a combination of those identified in the raw materials. Notably, the broad peak of amorphous silica observed in perlite raw materials disappeared after sintering, possibly due to the rearrangement of crystal structures during the high temperature process. The variations in sintering temperature did not induce a significant alteration in the composition of compounds within the samples. When comparing the XRD results, the pellets produced using MHH sintering exhibited comparable outcomes to those produced by conventional methods. This suggests that MHS sintering is a viable technique for achieving the desired crystalline structure in the ceramic pellets.

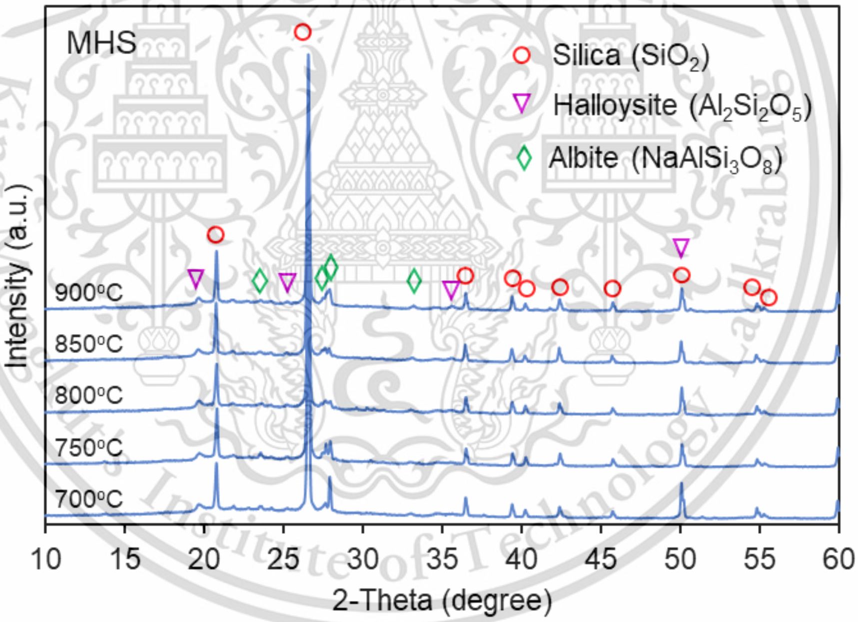


Figure 4.6 XRD patterns of clay pellets produced from microwave hybrid sintering at 700–900 °C.

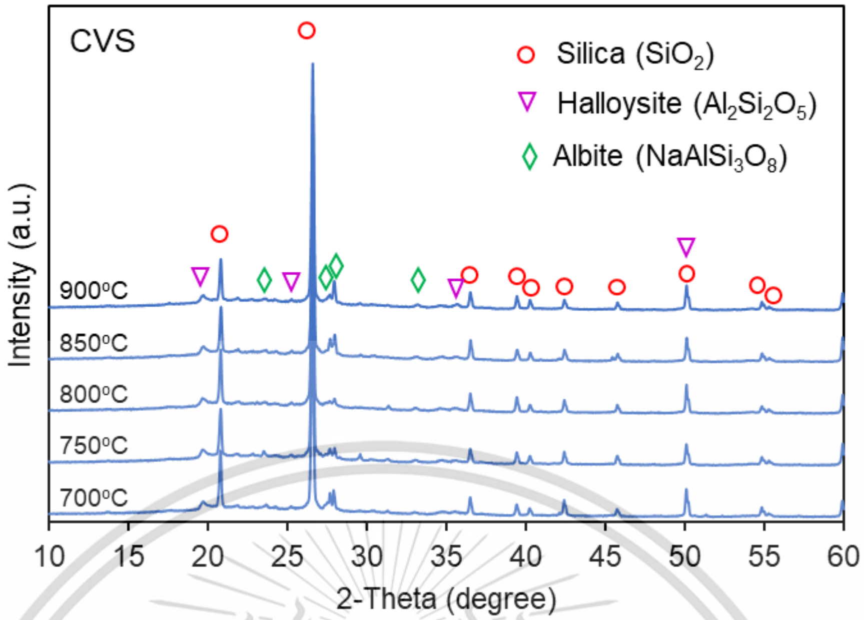


Figure 4.7 XRD patterns of clay pellets produced from conventional sintering at 700–900 °C.

4.2.2 Physical properties

Physical properties testing was conducted after the sintering of clay pellets using both microwave and conventional heating methods at temperatures of 700, 750, 800, 850, and 900 °C. The assessment included the determination of bulk density, porosity, and water absorption, utilizing the Archimedes method as described in section 3.4.1.

Figure 4.8 illustrates a comparison of bulk density between samples subjected to microwave hybrid sintering and conventional sintering. For clay pellets fired in both the microwave oven and electric furnace, the variation in bulk density remained consistent within the temperature range of 700 to 850 °C, while showed a slight increase at 900 °C. In the case of clay pellets sintered in the microwave oven, the bulk density ranged from 0.94 to 0.96 g/cm^3 at temperatures between 700 and 850 °C, reaching a maximum bulk density of 1.00 g/cm^3 at 900 °C. Meanwhile, clay pellets sintered with an electric furnace exhibited a bulk density in the range from 0.95 to 0.96 g/cm^3 at 700–850 °C, with the highest bulk density of 0.97 g/cm^3 at 900 °C.

Comparatively, clay pellets sintered using the microwave hybrid system demonstrated a slightly higher density. However, the density values for clay pellets from both methods are considered suitable for use as planting material due to their lightweight nature.

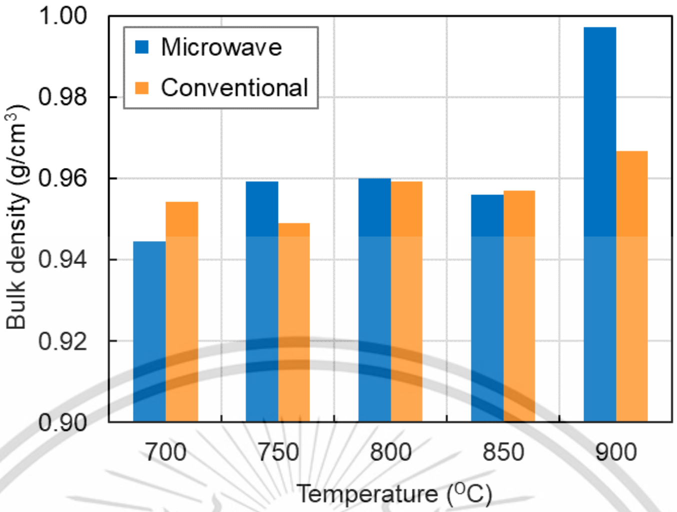


Figure 4.8 The bulk density of clay pellets produced from microwave hybrid heating and conventional sintering at 700–900 °C.

Figure 4.9 The results of porosity analysis for clay pellets sintered at temperatures ranging from 700 to 900 °C, using both microwave hybrid sintering and conventional sintering. The clay pellets sintered in a microwave oven exhibited a tendency to decrease in porosity, ranging from 48.4% to 43.5%, with increasing temperatures from 700 °C to 900 °C. Similarly, the porosity of clay pellets sintered with an electric furnace decreased from 48.4% to 46.9%, indicating a somewhat similar impact of sintering temperature on porosity values. In both sintering methods, the clay pellets exhibited the lowest porosity at 900 °C.

When comparing microwave-sintered clay pellets with those sintered using an electric furnace, the former displayed slightly lower porosity. Nevertheless, for all samples, the porosity values within this percentage range are deemed appropriate, facilitating sufficient air circulation in the soil for cultivation purposes.

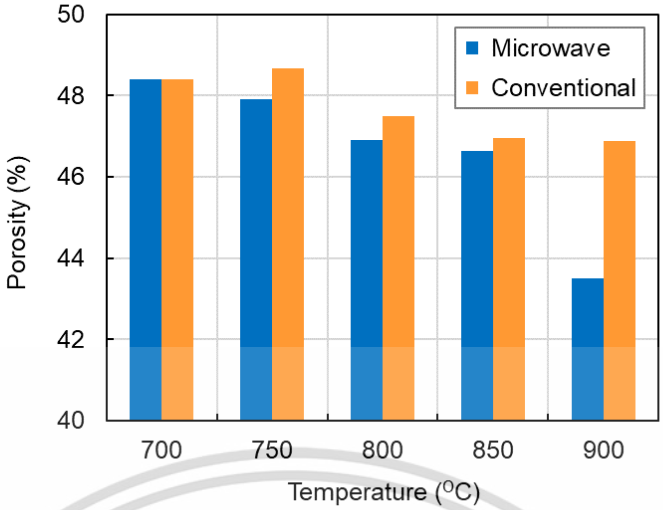


Figure 4.9 The porosity of clay pellets produced from microwave hybrid heating and conventional sintering at 700–900 °C.

Figure 4.10 demonstrates the results of examining the water absorption of clay pellets fired at temperatures ranging from 700 to 900 °C using both microwave hybrid sintering and conventional sintering. In both cases, there was a tendency for water absorption to slightly decrease as the sintering temperature increased.

The water absorption of clay pellets fired in a microwave oven decreased from 50.9% to 48.6% as the sintering temperature increased from 700 °C to 900 °C. Similarly, the water absorption of clay pellets fired in an electric furnace decreased from 51.3% to 43.9% within the same temperature range. Clay pellets sintered in a microwave oven exhibited slightly lower water absorption than those fired in an electric furnace. However, the water absorption remained within the appropriate range for absorbing water and mineral nutrients for plant growth.

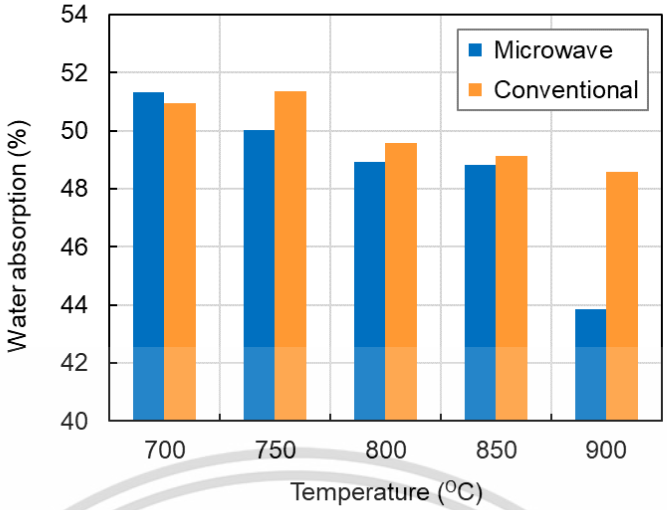


Figure 4.10 The water absorption of clay pellets produced from microwave hybrid heating and conventional sintering at 700–900 °C.

When comparing the sintering of clay pellets containing 30 wt% perlite using microwave hybrid sintering and the conventional sintering with an electric furnace at temperatures ranging from 700 to 900 °C, it was observed that the clay pellets exhibited similar physical properties. For instance, at 800 °C, clay pellets sintered using a microwave hybrid system showed a density of 0.96 g/cm^3, porosity of 46.9%, and water absorption of 48.9%. In comparison, clay pellets sintered with a conventional furnace showed a density of 0.96 g/cm^3, porosity of 47.5%, and water absorption of 49.6%.

Notably, the microwave hybrid sintering system demonstrated a significant efficiency advantage, completing the sintering process approximately five times faster. This suggests that microwave hybrid sintering is advantageous in terms of both speed and energy economy. Table 4.1 provides a summary of the physical properties of commercial sintered pellets compared to those produced using microwave hybrid sintering (MHS). Commercial sintered pellets exhibit a wider range of bulk density, porosity, and water absorption. The physical properties of pellets sintered using microwave hybrid sintering fall within the range of those of commercial pellets prepared using conventional sintering processes. Therefore, microwave hybrid sintering can achieve comparable results more efficiently and potentially more economically.

Table 4.1 Summary of physical properties of commercial sintered pellets compared to microwave hybrid heating.

Planting pellets	Bulk density (g)	Porosity (%)	Water absorption (%)
Commercial	0.47 – 1.59	38.5 – 65.0	22.4 – 138.5
MHS	0.94 – 1.00	43.5 – 48.4	48.6 – 50.9

4.2.3 Microstructure of sintered pellets

The microstructures of the cross-sectional surfaces of clay pellets produced by microwave hybrid sintering (MHS) and conventional sintering (CVS) at various temperatures are displayed in Figures 4.11 and 4.12, respectively. In those images, the white particles representing perlite are homogeneously distributed within the clay pellet matrix. There is no discernible difference between the microstructures of the pellets produced by MHS and CVS, and variations in temperature do not appear to affect the microstructure significantly.

The homogenous distribution of perlite within the clay matrix indicates that both sintering methods, MHS and CVS, effectively integrate the perlite into the clay. This uniform distribution is crucial for maintaining the structural integrity and performance characteristics of the pellets. The lack of significant microstructural differences between the two methods suggests that MHS can achieve comparable results to CVS, even though MHS operates more quickly and efficiently.

Additionally, the observation that different temperatures do not markedly affect the microstructure highlights the robustness of both sintering processes. This stability across temperatures ensures that the physical properties of the pellets remain consistent, which is beneficial for applications requiring reliable material performance. Therefore, MHS presents a viable alternative to CVS, offering advantages in speed and energy efficiency without compromising the quality of the final product.

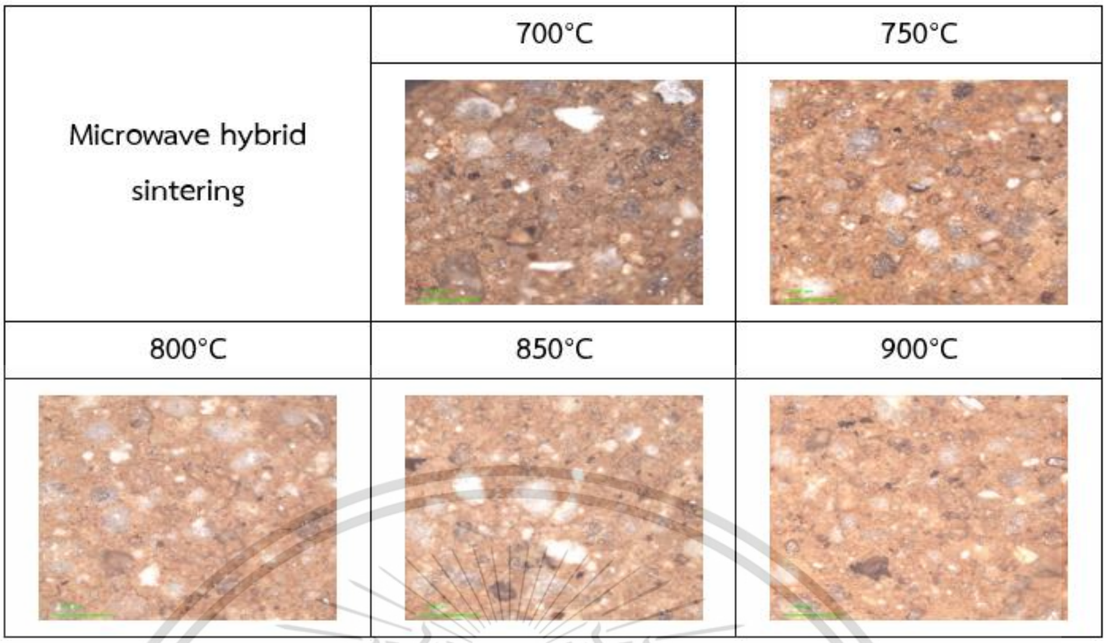


Figure 4.11 The microstructures of the cross-sectional surfaces of clay pellets produced by microwave hybrid sintering (MHS).

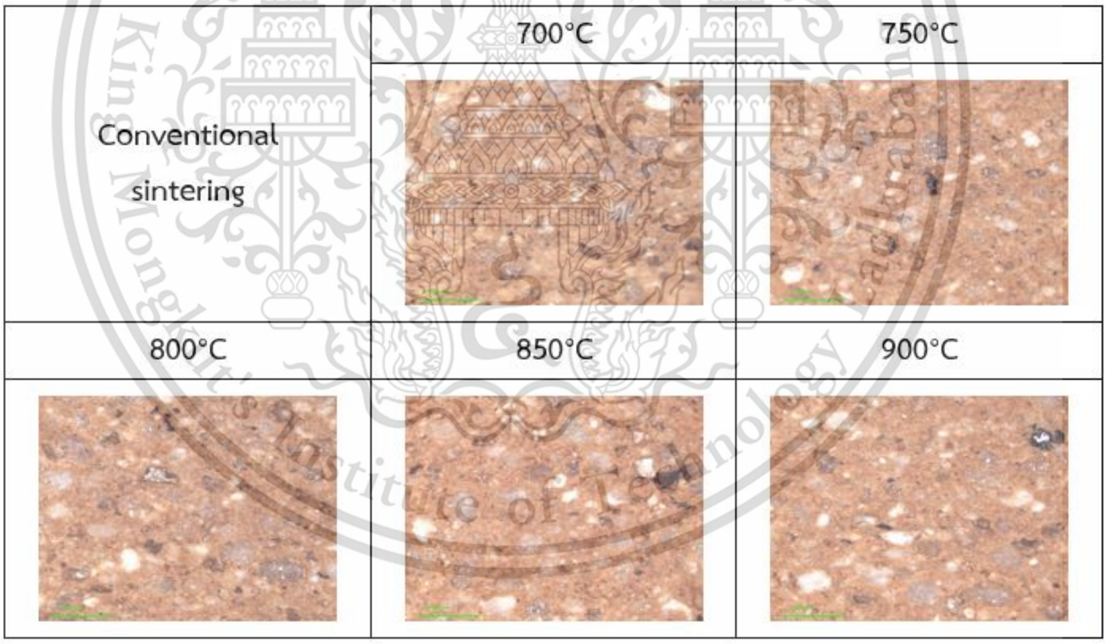


Figure 4.12 The microstructures of the cross-sectional surfaces of clay pellets produced by conventional sintering (CVS).

4.2.4 Mechanical properties

The crushing strength (σ_c) of the clay pellets was measured to observe the effects of sintering temperature and sintering method. The results, displayed in Figure 4.13, indicate that the crushing strength of clay pellets fired in a microwave oven rapidly increased from 1.04 MPa to 3.78 MPa as the sintering temperature rose from 700°C to 900°C. In contrast, the crushing strength of clay pellets fired in an electric furnace showed a slower increase, from 0.99 MPa to 1.21 MPa within the same temperature range. The crushing strength of samples sintered by both methods increased with higher sintering temperatures. However, in conventional sintering, the difference in crushing strength across various temperatures was minimal. This suggests that microwave sintering is more effective in enhancing the mechanical strength of the clay pellets compared to conventional electric furnace sintering, likely due to the more uniform and rapid heating provided by microwave energy.

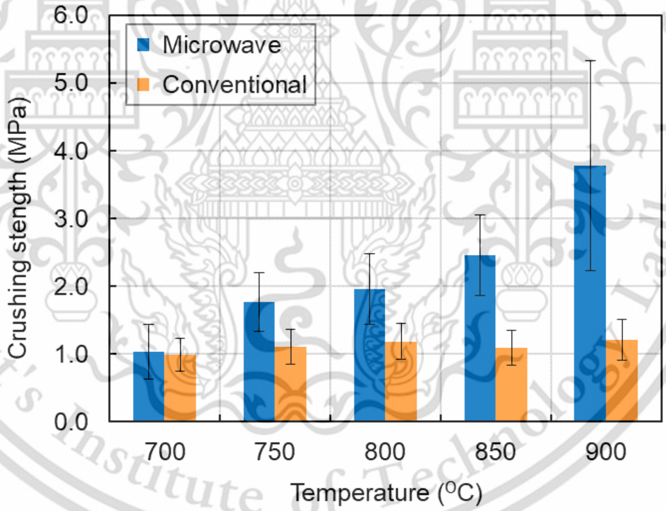


Figure 4.13 The crushing strength of clay pellets produced from microwave hybrid heating and conventional sintering at 700–900 °C.

After the crushing strength test, the crack behavior of clay pellets was observed. The photo of fracture pattern on clay pellets sintered by conventional sintering and microwave hybrid sintering are shown in Figure 4.14 and Figure 4.15, respectively. The fracture patterns illustrate the differences between conventional sintering and microwave hybrid sintering across various temperatures. In conventional sintering, at 700°C to 900°C, the fracture patterns remain relatively consistent, showing small cracks without significant variation. This indicates that the structural integrity of the pellets remains stable, with minor increases in internal stress and densification as temperature increases.

Conventional sintering	700°C	750°C
		
800°C	850°C	900°C
		

Figure 4.14 The fracture pattern in sample sintered by conventional sintering.

For microwave hybrid sintering, at 700°C to 750°C, the fracture patterns are similar to those seen in conventional sintering, with small cracks appearing on the surface. This suggests that at lower temperatures, the microwave hybrid method does not drastically alter the structural integrity of the pellets. At 800°C, significant cracks start to appear, indicating a marked increase in internal stresses. This is likely due to rapid heating and uneven temperature distribution, which causes differential expansion and contraction within the pellet. At 850-900°C, the cracks become more severe, with some pellets splitting into multiple pieces. This severe cracking pattern corresponds with the observed increase in crushing strength, suggesting that while the microwave hybrid sintering method enhances densification and mechanical strength, it also induces significant internal stresses that compromise structural integrity.

The increased crushing strength in microwave hybrid sintering correlates with more severe cracking patterns, particularly at higher temperatures. This indicates that the rapid heating process enhances densification but also introduces substantial internal stresses, leading to structural failure.

Microwave hybrid sintering	700°C	750°C
		
800°C	850°C	900°C
		

Figure 4.15 The fracture pattern in sample sintered by microwave hybrid sintering.

Chapter 5

Conclusion

In the study of the susceptor, it was found that microwave power has a direct effect on the heating rate. However, adding more SiC susceptors does not necessarily increase the heating rate. Specifically, this study found that two plates of SiC susceptors offered a higher heating rate than three plates. Adding more susceptors decreases the penetration depth of the microwave because excessive absorbers increase the reflection of microwaves, creating low-intensity areas due to the standing wave effect. Consequently, the most effective condition identified in this study is using two plates of SiC with the microwave power set at 100%, achieving a heating rate of 62°C/min.

The study compares the sintering of clay pellets using conventional heating and microwave hybrid heating. XRD patterns of samples sintered by both methods revealed the same phases of compounds, indicating that the sintering temperature did not significantly affect the phase composition. However, the properties differed between the two methods. Clay pellets sintered with microwave hybrid heating exhibited higher bulk density, but lower porosity and water absorption compared to those sintered using conventional methods. The mechanical properties of samples sintered with conventional heating showed no significant effect of sintering temperature, with crushing strength ranging from 0.99 to 1.20 MPa. In contrast, samples sintered with microwave hybrid heating showed a notable increase in crushing strength, rising from 1.04 MPa to 3.78 MPa at 700-900°C.

Overall, microwave hybrid heating improved the mechanical properties of the clay pellets and achieved a heating rate five times faster than conventional heating, leading to significant time and energy savings. The optimal sintering conditions were identified as 900°C using MSH, resulting in clay pellets with a density of 1.00 g/cm³, porosity of 43.5%, water absorption of 43.9%, and a noteworthy crushing strength of 3.78 MPa.

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Appendix

A STUDY ON SiC SUSCEPTOR CONFIGURATION FOR MICROWAVE HYBRID HEATING

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Abstract

Microwave hybrid heating (MHH) is a novel method to enhance ceramic sintering at high temperatures. The heating mechanism by MHH involves two directions of heat transfer for materials: microwaves heat the sample from the inside out, while the susceptor provides conventional heating from the outside. This unique heating mechanism offers several advantages, including uniform heating, rapid sintering, and enhanced microstructure and properties of materials. This study investigates the configuration of silicon carbide (SiC) susceptors for microwave hybrid heating. The microwave oven (multi-mode, 2.45 GHz, 1.2 kW) was modified with a ceramic insulator housing to maintain the temperature in the chamber. The effects of different configurations of SiC susceptors and microwave powers on the heating rate and maximum temperature were investigated. SiC susceptor plates were placed in the microwave oven using 3 different configurations, and for each condition, the microwave power was varied at 40, 60, 80, and 100% (480, 720, 960, and 1,200 W). The temperature in the microwave chamber was recorded until it reached 900°C or after 30 min of heating. Using two plates of SiC susceptor at 100 % power resulted in the highest heating rate of 62°C/min to reach 925°C. The results of this study offer guidance for the selection of appropriate heating conditions for individual ceramic materials, which can lead to more effective sintering processes.

Keywords: Microwave hybrid heating; microwave sintering; silicon carbide; susceptor; SiC susceptor

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Introduction

Microwave technology has shown promising prospect in the field of materials processing, particularly in ceramic sintering. Compared to conventional sintering methods, microwave sintering offers advantages such as reduced processing time, improved microstructure and mechanical properties, and energy efficiency (Madhan and Prabhakaran, 2019; Lyra *et al.*, 2019; Zeng *et al.*, 2019; Guillén *et al.*, 2021).

However, certain types of ceramic materials may not be able to absorb microwave energy effectively, leading to low sintering temperatures. Furthermore, microwave heating can result in temperature differences between the core and surface of the material due to the heat being primarily generated within the material (Bhattacharya and Basak, 2016). To overcome these issues, microwave hybrid heating (MHH) techniques have been developed. These methods incorporate a susceptor, which absorbs microwave energy and converts it to heat, allowing for both microwave and conventional heating to occur simultaneously. MHH process allows for the sample to be heated from the inside out via microwaves, while the susceptor provides conventional heating from the outside in. This technique has been shown to enhance the heating rate and provide more uniform heating of the material, resulting in improving of sintered products (Huang *et al.*, 2009; Bhattacharya and Basak, 2016). The utilization of MHH techniques has the potential to significantly improve the efficiency of ceramic sintering processes, leading to advances in the development of new materials with superior properties.

The interaction of materials with microwaves is an important factor in the success of microwave heating for materials processing. The ability of a material to absorb microwave energy and convert it into heat is determined by its penetration depth (d_p) and loss tangent ($\tan\delta_e$) value (Mishra and Sharma, 2016). A material with a high penetration depth (d_p) can absorb microwave energy more effectively, while a high $\tan\delta_e$ value indicates the ability to convert this energy into heat. An effective susceptor material should possess both these parameters, with a high penetration depth and a high value of $\tan\delta_e$. The selection of an appropriate susceptor material is thus important in achieving successful microwave hybrid heating of ceramic materials.

Table 1 presents the penetration depth (d_p) and loss tangent ($\tan\delta_e$) of microwave absorber materials (Bhattacharya and Basak, 2016; Mishra and Sharma, 2016). Carbon materials, such as carbon black, graphite and activated carbon, have a high value of $\tan\delta_e$ and a penetration depth in the order of centimeters. However, carbon tended to decompose at temperatures above 600°C, which renders them unsuitable for used as susceptor at high temperatures. On the other hand, silicon carbide (SiC) has excellent penetration depth (d_p) and loss tangent ($\tan\delta_e$) and is highly stable at high temperatures. SiC demonstrates excellent suitability as a susceptor in microwave hybrid heating systems due to its high melting point of approximately 2,470°C (Taneja *et al.*, 2021). Moreover, SiC exhibits remarkable stability, as oxidation typically occurs at much higher temperatures, around 1,300-1,600°C (Chen *et al.*, 2021). Therefore, SiC is a good candidate for use as a susceptor for high temperature application (Huang *et al.*, 2009; Satapathy, 2009; Yahaya *et al.*, 2013; Bhattacharya and Basak, 2016; Bhoi *et al.*, 2019).

The efficiency of heating in MHH process is dependent on the design of susceptor. To achieve efficient heating, the susceptor should provide pre-heating to the material to allow for uniform heating throughout the sample, but it should not completely block the microwave energy from reaching the material. The amount and position of the susceptor can also impact the efficiency of heating. A larger amount of susceptor material can absorb more microwave energy, but it can also block more of the energy from reaching the material being heated. The position of the susceptor can also impact the efficiency, with a well-designed susceptor configuration allowing for efficient heating of the material.

The design of the susceptor is an important consideration in achieving efficient microwave-assisted heating in MHH processes. The objective of this study is to examine the parameters of a microwave hybrid heating system, including microwave power, number of susceptors, and susceptor layout. The experiment was conducted utilizing a modified microwave oven to investigate

Table 1. The penetration depth (d_p) and loss tangent ($\tan\delta_e$) of microwave absorber materials (Bhattacharya and Basak, 2016; Mishra and Sharma, 2016)

Materials	d_p (cm)	$\tan\delta_e$
SiC	1.93	0.37
Carbon black (20 μm)	5.75	0.23
Graphite powder (20-80 μm)	1.34–2.09	0.36–0.67
Activated carbon	0.7–3.43	0.31–0.9
Charcoal	6–11	0.14–0.38
Carbon fiber	0.5–0.7	0.45–0.5
Carbon nanotube	0.2	1.11

the most efficient condition to employ SiC susceptors in microwave hybrid heating.

Experimental Methods

In this study, microwave oven (Sharp, model R-390, multi-mode, 2.45 GHz, 1.2 kW) was utilized. Additionally, an alumina-silica ceramic fiber board housing was inserted inside the microwave chamber as an insulator to prevent heat loss.

SiC plate used as susceptor in this experiment has a thickness of 1.2 cm. The SiC plate was cut to 2 sizes. The dimension of SiC plates that was placed at left- and right-side of the sample was 8.5 cm × 6.0 cm in height × width. On the other hand, the SiC plate placed at the bottom of the sample had dimensions of 10 cm × 10 cm in height and width. To evaluate efficiency of the microwave hybrid heating, the experiment was designed in three different configurations, each with a different number of SiC plates, as displayed in the Figure 1.

Furthermore, different microwave power levels to identify the most efficient power level for sintering was examined. For this experiment, microwave powers at 20, 40, 60, 80, and 100% (240, 480, 720, 960, and 1,200 W) were utilized. The temperature within the microwave oven was measured using a Type K thermocouple. The thermocouple probe was carefully inserted at the top of the microwave oven, ensuring that it was positioned near the sample being heated. The test was conducted for 30 min or until the temperature reached 900°C.

Result and Discussion

An investigation was conducted to examine the correlation between the number of SiC susceptor plates, the electrical power of a microwave oven, and the resulting temperature inside the microwave cavity. According to Table 1, SiC has a high penetration depth of 1.93 cm and a high loss tangent value of 0.37. The SiC susceptor used in this study had a thickness of 1.2 cm. Therefore, microwave energy can effectively penetrate the susceptor, allowing for efficient heating. From the experiment results, it was found that the temperature inside the microwave oven increased with time, and an increase in electrical power also resulted in an increase in temperature.

Figure 2 shows the temperature profiles in the microwave oven when using one SiC susceptor

Figure 1. The configuration of silicon carbide susceptor plates inside the microwave: (a) one susceptor plate, (b) two susceptor plates, and (c) three susceptor plates

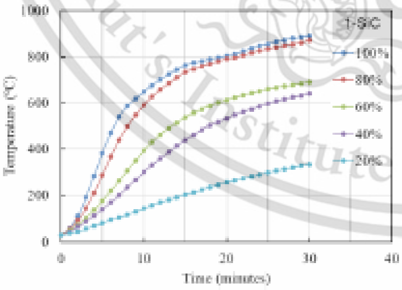

Figure 2. Temperature profiles in the microwave oven when using one SiC susceptor plate, with microwave powers at 20, 40, 60, 80, and 100%

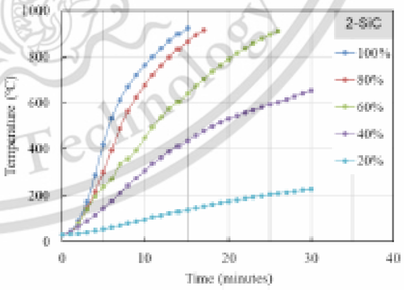

Figure 3. Temperature profiles in the microwave oven when using two SiC susceptor plates, with microwave powers at 20, 40, 60, 80, and 100%

plate. With a single SiC susceptor and 20% electrical power, the highest temperature reached only 334°C after 30 min. Upon increasing the electrical power to 40% and 60%, the maximum temperature rose to 640°C and 689°C respectively, after 30 min. Similarly, when increasing the electrical power to 80% and 100%, the temperature followed a similar increasing pattern, with maximum temperatures of 870°C and 889°C, respectively, after 30 min. However, using only one SiC susceptor plate resulted in a slower rate of temperature increase compared to using two SiC susceptors.

Figure 3 shows the temperature profiles in the microwave oven when using two SiC susceptor plates. When the electrical power was set at 20% and held for a duration of 30 min, the maximum temperature reached was only 226°C. Subsequently, when the electrical power was increased to 40%, the maximum temperature increased to 650°C. Further increasing the electrical power to 60%, 80%, and 100% resulted in a rapid temperature rise, reaching a maximum of 900°C before the 30 min. The applying of 60% electrical power resulted in the temperature inside the oven reaching 912°C at 26 min, while 80% electrical power achieved a temperature of 915°C at 17 min. Finally, the highest temperature of 925°C was attained within only 15 min by increasing the electrical power to 100%.

Figure 4 illustrates the temperature profiles in the microwave oven when using three SiC susceptor plates. When applying 20% electrical power, the maximum temperature reached was 314°C. Increasing the electrical power to 40% and 60% resulted in maximum temperatures of 591°C and 888°C, respectively. Applying 80% electrical power resulted in a maximum temperature of 888°C. Finally, when using 100% electrical power, the maximum temperature achieved inside the oven was 913°C after 30 min.

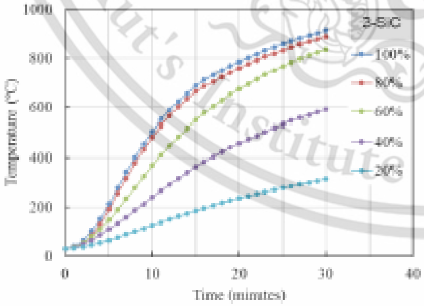

Figure 4. **Temperature profiles in the microwave oven when using three SiC susceptor plates, with microwave powers at 20, 40, 60, 80, and 100%**

It was observed that increasing the number of SiC susceptor from two plates to three plates did not result in an increase in the heating rate. Conversely, the use of three susceptor plates resulted in a decrease in the heating rate. This reduction in efficiency can be attributed to wave interference and reduced microwave energy absorption caused by an excess of susceptors. The presence of too many susceptors can impede the propagation of microwaves, causing standing waves and a reduction in penetration depth. In microwave heating, standing waves can occur when there is interference between the waves reflected from different surfaces within the oven. When microwaves encounter a reflective surface, such as a metal or SiC susceptor, they can be reflected back towards the source. If these reflected waves combine with incoming waves, they can create areas of high and low intensity that remain fixed in space, leading to standing wave patterns. Therefore, this result in this research clearly indicated that it is important to carefully optimize the number and placement of susceptors in order to achieve high heating efficiency in microwave hybrid heating.

In this research, the optimal heating conditions for microwave hybrid heating (MHH) were investigated, and it was found that using two silicon carbide susceptor plates provided the most efficient heating. By applying microwave power at 100% (1,200 W), the oven temperature reached a maximum of 925°C in 15 min. This heating rate averaged at 62°C/min, which is comparatively higher than other conditions. In addition to the observed effects of the number of SiC plates and microwave power levels on temperature, the position of SiC within the chamber has a significant impact on the temperature distribution in the microwave hybrid heating system due to variations in microwave absorption and reflection angles. In future research, it is important to examine the effect of the position of SiC susceptor on the temperature profile within the chamber.

Conclusions

This study investigated the effectiveness of different configurations of silicon carbide (SiC) susceptors in microwave hybrid heating (MHH) for sintering ceramics. The results suggest that the use of two SiC susceptor plates with microwave power at 100% (1,200 W) is the most effective configuration for achieving a high heating rate of 62°C/min to reach the target temperature of 925°C within 15 min. It was also observed that increasing the number of SiC susceptors (three plates) led to a decrease in heating rate due to the impedance of microwave absorption. The findings of this study provide guidance for selecting appropriate heating conditions to enhance the sintering process and improve the properties of materials. Further studies can explore the applicability of MHH for sintering of various kinds of material and optimizing the heating parameters for specific applications.

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