

# Geopolymers Prepared by Microwave Treatments



Yuta Watanabe and Takaomi Kobayashi

**Abstract** Geopolymers occur easily in a polycondensed polymer framework in high-alkali conditions. They are known as sustainable materials with a simple process that uses waste for raw materials like fly ash, slag, and rice husk etc. This chapter highlights the use of microwaves as efficient auxiliary processing for geopolymer production. Compared to conventional heating, the microwave cure reduced the curing time as an advantage. In addition, depending on the microwave irradiation conditions, the resulting geopolymers were changed in material properties such as high porosity, and low mechanical strength using a high-water foaming agent content due to water evaporation in the geopolymer. Also, the microwave curing had the advantage over the conventional oven heating, of densely structuring geopolymers when the water foaming agent content was low by combining the pre-heating and accelerating the polymerization. In contrast, the alkali activator concentration caused enhancement of the morphology of geopolymers in the case of microwave cure. Thus, microwave curing is expected to be an effective method for the fabrication of geopolymers. This chapter describes the feasibility of synthesizing the porous morphology and densely structured geopolymer without micro-sized pores using microwave energy.

**Keywords** Geopolymer · Microwave · Alkali activator · Silica-aluminum linkage · Replacement concrete

## 1 Overviewing Geopolymers

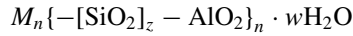
Geopolymers are usually activated in highly alkaline solutions and aluminosilicate geopolymers are inorganic materials consisting of tetrahedral  $\text{AlO}_4$  and  $\text{SiO}_4$  units [1, 2]. Essentially a mineral chemical compound or mixture of compounds consist of repeating units, for example, silico-aluminate (polysialate) ( $-\text{Si}-\text{O}-\text{Al}-\text{O}-$ ) and is created through a process of polymerization. The polymerization process involves

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chemical reactions under highly alkaline solution conditions in Si and Al materials that provide covalent Si–O– and Al–O bonds, with the following formula [3].



where  $M$  is an alkali cation such as sodium ( $\text{Na}^+$ ) or potassium ( $\text{K}^+$ ),—refers to bonding,  $n$  is the polymerization degree,  $z$  is usually a number of 1, 2, 3 and sometimes higher, and  $w$  is the number of moles of water. When strong alkalis activate the silicon and aluminum in the raw materials, geopolymerization allows transforming glassy structure partially or totally into a very compact composite [3, 4]. In these processes, as shown in Fig. 1, the alkali activator accelerates the condensation reaction, then leaches to form alumino-silicate precursor in the aqueous solution and finally the condensates to be geopolymer gels and then progressing polymerization. As a result, the mixture becomes hardened geopolymer [3, 5]. The geopolymer network generally proceeds at moderate temperature below 160 °C. For the Si and Al materials, generally alumino-silicates such as slag [6], metakaolin [7, 8], and fly ash [9, 10] were mixed with high concentration of alkaline solution at low temperatures. For example, NaOH and KOH are mainly used as highly concentrated alkalis, and the aforementioned materials containing Si and Al become soluble in alkaline solutions [1, 3, 11]. Here, Si and Al rich materials are the primary requirement for geopolymerization like rice husk [12, 13], clays like metakaolin [14, 15], natural soil [16] and bentonites [17, 18] as seen in Table 1. Geopolymers can be made from several types of alumino-silicate materials, including metakaolin, calcined clay [15], agricultural waste ashes [12, 13], and industrial sludge waste [19]. In the waste, the geopolymers based on industrial waste like slags [20–22], and aluminum waste [23] were helpful in the geopolymer fabrication.

Looking at the content of  $\text{Al}_2\text{O}_3$  and  $\text{SiO}_2$  in Table 1, the wastes contain the necessary components for geopolymer. In the cases of agricultural waste like rice husks, the ashes involve high silica contents depending on sintering temperature. The higher temperature the higher silica contents remain like about 90%, although

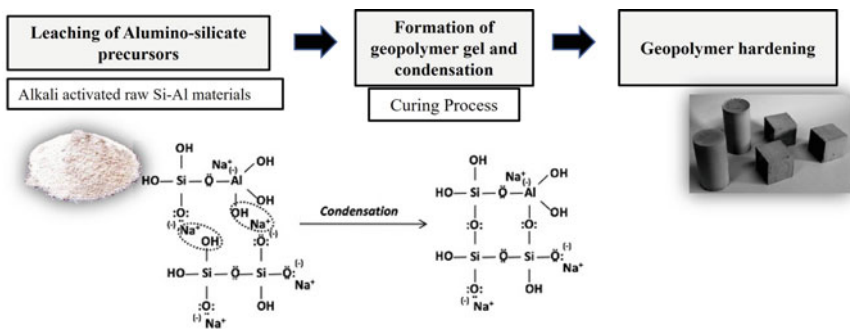


Fig. 1 Scheme of geopolymerization of silica alumina precursor by geopolymerization process

**Table 1** Chemical compositions of raw materials for synthesis geopolymers

Compositions (%)	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	Na <sub>2</sub> O	MgO	P <sub>2</sub> O <sub>5</sub>	SO <sub>3</sub>	K <sub>2</sub> O	CaO	TiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	References
Rice husk*	0.09	92.19	1.64	0.41	0.01	0.41	0.05	0.09	0.71	0.1	[12]
Rice husk**	1.4	76.8	–	0.5	0.6	0.7	3.7	0.5	–	0.1	[13]
Metakaolin	42.63	50.00	0.28	0.13	–	0.44	0.34	1.29	1.71	2.11	[14]
Na-bentonite	15.79	68.28	2.57	4.08	0.04	0.02	0.87	5.5	0.24	2.29	[18]
Kaolin	43.98	53.39	–	0.08	–	0.01	0.86	0.04	–	0.63	[19]
Calcined kaolin waste	33.85	56.56	0.23	0.05	–	–	5.15	0.05	0.16	2.16	[15]
Fly ash***	18.37	31.94	1.2	1.86	0.16	5.13	3.05	19.84	0.4	17.39	[19]
Aluminum hydroxide waste	63.28	0.18	–	0.31	0.01	<0.01	0.01	0.01	0.01	–	[10]
GCBS****	33.6	12.9	0.3	7.9	–	2.2	0.4	41.5	0.7	0.6	[22]

\* Incinerating at 650–700 °C for 1 h in India

\*\* Calcined at 400 °C for 10 h in Taiwan

\*\*\* Supplied from Mae Moh power plant in Lumpang (Thailand)

\*\*\*\* Granulated Blast-Furnace Slag (from volcanic rocks)

alumina contents are less. In this case, metakaolin was used as alumina source, and the mixture with rice husk ash enabled the geopolymerization [8, 12].

Among these starting materials for geopolymer, fly ashes are interesting from the perspective of sustainable materials. Thus, fly ash-based geopolymer is an important material because such ash is the one of bulky wastes resulting from burning coal or lignite in power generation facilities. For using an environmentally friendly method [20, 21], fly ash of chemical composition of silica and alumina typically ranging from 40 to 60%, and from 20 to 30%, respectively is available worldwide for a geopolymer source. Thus, the condensated aluminosilicate is known as an eco-friendly material due to recycling of burnt ash, and another advantage is obtaining materials at low temperature for the polycondensation, which easily occurs under high alkaline conditions. In various types of slags used individually or as a partial replacement of fly ash, lightweight geopolymers have been obtained using palm oil fuel ash [23], ground granulated blast furnace slag [24], low calcium fly ash [25], and class F fly ash, ground granulated blast furnace slag [26] and metakaolin/biomass ash composite [27]. In the geopolymer preparation processes, heating and ambient curing condition were investigated using sodium silicate [28], alkali activator [9, 15, 29], curing temperature as sintering condition [7, 30] and additives [31]. Geopolymer made from industrial waste and by-products is low cost and environmentally friendly from environmental point of view. Therefore, scientists are still researching the many possible uses of geopolymer processes. In addition, geopolymers have various applications, and their properties can be engineered. Some papers reported that geopolymers in general have material structure with high apparent bulk density in the range of over  $1 \text{ g/cm}^3$  causing high mechanical strength [10]. On the other hand, when geopolymer bulk has pores using foaming agents in the preparation, in general, the density and mechanical strength decrease. In the latter cases with pore structure, the bulk had a high specific surface area and high porosity, therefore, such geopolymers were expected to apply to adsorbents [8, 32–34].

There are disadvantages of geopolymers as compared to traditional cement in certain aspects like long maintenance time and slow curing till enough strength development, becoming the limiting factor for practical applications of geopolymers. However, geopolymers are interesting in technologies that enable the rapid development of strength in the fields. The common geopolymer products use conventional thermal curing under water, air, solar, or oven curing conditions to accelerate geopolymerization process. Table 2 summarizes advantages and disadvantages of each curing method of geopolymers. As seen in Table 2, water-cured geopolymer was found to be of lower compressive strength than air-cured, solar- and oven-cured. Water curing had disadvantages of reducing strength due to leaching reactant of geopolymerization and alkali activator solution [35]. Air and solar-cured geopolymer could be of higher compressive strength than the one cured with other conventional curing methods, these curing methods had the advantages that curing with both of them needs low energy source, and decreases manufacturing cost and carbon footprint. However, there were still limitations on the application of these curing methods due to the long curing time (air: 7–90 days, and solar: 3–28 days), even though these changes depend upon condition and geological location [36, 37]. Traditionally, oven heating was used

as the most effective method for producing geopolymers with higher compressive strength, because of easy adjustment of the curing temperature and producing at 60–120 °C for 6–24 h in relatively a short time and low temperature [4, 10, 22]. However, among the conventional curing methods, oven curing especially has high heat loss due to the distribution of heat in the geopolymer from the exterior to the interior leading to the non-uniform [38]. Moreover, it was necessary for long heating within 6–24 h and high energy consumption for the curing. In addition, a limitation was the use of a foaming agent for a porous geopolymer. On the other hand, microwave curing could be attractive method for geopolymerization in recent years. Compared with the conventional heating methods, microwave curing by standardized volumetric measurement has some advantages of heating as shown below.

- (1) Shorter curing time for geopolymerization (1–60 min) [39–42].
- (2) Easier operation for fabrication of geopolymers [39, 43].
- (3) No requirement of foaming agents for porous morphology geopolymers [16, 39].

## 2 Effect of Microwave on Geopolymerization

Recently, from the perspective of reducing energy consumption and humbleness, microwave energy has been utilized as an innovative tool for heating to influence chemical reactions of geopolymerization [39–41, 43, 44]. This method includes the benefits of microwave heating; higher heating rates in less times, no direct contact between the reactants and energy source, and clean, selective, and remote heating of the reactants in the desired atmosphere [45]. Microwave method utilized the bottom ash to produce thermal insulating bricks for foaming of the mortar by geopolymerization [44].

Using microwave radiation, the conventional heating was helpful for obtaining geopolymers attaining thermal stability as the dense matrices. Nevertheless, reports are few in the literature about microwave cured geopolymers. But, with these benefits, microwave-assisted synthesis of concrete [46, 47] and zeolite [48–50] were known to be the effectiveness, because the activation time in hydrothermal synthesis was reduced automatically. Table 3 lists researches of microwave cure for geopolymerization. There are two types of geopolymers in the dense and porous conformation.

It is known that the conventional heating has been used as the popular way to accelerate the geopolymerization, but heat is distributed in the sample from the exterior to interior leading to the non-uniform and long curing time [38, 40]. Then, microwave consisting of an electric and a magnetic field component has the ability to penetrate deep into the materials in nature within a short time. Samples containing inorganic ions that absorb microwave energy can heat volumetrically [51]. The electric component of an electromagnetic field causes heating by two main mechanisms: dipolar polarization and ionic conduction [45, 52, 53]. Under microwave condition

**Table 2** Advantages and disadvantages of each curing method for geopolymer fabrication

Curing method	Water [42, 35]	Air [37]	Solar [36]	Oven [4, 10, 22]	Microwave [39, 39–42]
Raw materials	Fly Ash + Slag	Fly Ash	Fly Ash + Ground Granulated Blast-Furnace Slag (GGBS)	Fly Ash, GCBS	Fly Ash, Natural Soil
Reaction temperature	23 °C	Room temperature (22–25 °C)	20–60 °C	60–120 °C	40–200 °C
Curing time	Long (1–28 days)	Very long (7–90 days)	Very long (3–28 days)	Short (6–24 h)	Very short (1–60 min)
Morphology	Dense	Dense	Dense	Dense	Dense, Porous
Compressive strength (MPa)	10–48 MPa	30–65 MPa	25–90 MPa	20–70 MPa	Dense: 30–90 MPa  Porous: 3.1–8.8 MPa
Advantages	Small temperature changes	Low energy force	Decreasing the manufacturing cost and carbon footprint	Simple operation	Short time curing
				Easy controlling the curing temperature	Easy operation
Disadvantages	Leaching of alkaline activator	Long curing time	Depending on weather condition and geographical location	High energy requirements	No requirement of foaming agents for porous morphology Difficulty in reaction controlling
		Depending on the environment temperature		High heat loss	

**Table 3** Researches of microwave cure for geopolymerization

Raw materials	Morphology	Microwave curing (MWC) conditions	Geopolymerization conditions	Chemical composition (XRF)	Compressive strength (CS)	Density	Reference
Fly ash (less than 10% Ca component)	Dense morphology	Pre-curing at ambient temperature + MWC 240,360 and 600W MW for 120 min after ambient curing for 12 h	NaOH: 4, 6, 8 and 10 M River sand	SiO <sub>2</sub> : 50.2%, Al <sub>2</sub> O <sub>3</sub> : 28.7%, Fe <sub>2</sub> O <sub>3</sub> : 5.72%, CaO: 5.9%, MgO: 1.7%, Na <sub>2</sub> O: 1.0%, SO <sub>3</sub> : 1.0%	56 MPa (MWC at 240W for 120 min, with 10 M-NaOH)	–	[43]
Fly ash (Higher than 10% Ca component)		MWC + Oven cure 90W MWC for 5 min + oven cure at 65 °C for 3,6 and 12 h	NaOH: 10 M Na <sub>2</sub> SiO <sub>3</sub> Sand (The mass ratio of Na <sub>2</sub> SiO <sub>3</sub> /NaOH was 1.5)	SiO <sub>2</sub> : 39.2%, Al <sub>2</sub> O <sub>3</sub> : 19.7%, Fe <sub>2</sub> O <sub>3</sub> : 12.1%, CaO: 16.9%, MgO: 1.7%, Na <sub>2</sub> O: 1.9%, SO <sub>3</sub> : 2.9%	42.5 MPa (MWC at 90W for 5 min + Oven cure at 65 °C for 12 h)	–	[41]
Granulated blast-furnace slag (GCBFS)		Pre-curing at ambient temperature + MWC 350, 540 and 750W Pre-curing for 3 days + MWC for 5 min	NaOH Na <sub>2</sub> SiO <sub>3</sub> (Alkali modulus of Na <sub>2</sub> O/SiO <sub>2</sub> was 1.25)	SiO <sub>2</sub> : 33.6%, Al <sub>2</sub> O <sub>3</sub> : 12.9%, Fe <sub>2</sub> O <sub>3</sub> : 0.6%, CaO: 41.5%, MgO: 7.9%, Na <sub>2</sub> O: 0.3%, SO <sub>3</sub> : 2.2%	43 MPa (pre-curing + MWC at 350W)	–	[22]

(continued)

Table 3 (continued)

Raw materials	Morphology	Microwave curing (MWC) conditions	Geopolymerization conditions	Chemical composition (XRF)	Compressive strength (CS)	Density	Reference
Metakaolin		Pre-curing at ambient temperature + MWC 3 kW MWC for 26 min used the second day at ambient temperature	Ca(OH) <sub>2</sub> Potassium water glass (Si/Al = 1.4–1.85)	SiO <sub>2</sub> : 52.0%, Al <sub>2</sub> O <sub>3</sub> : 41.5%, Fe <sub>2</sub> O <sub>3</sub> : 1.1%, CaO: 0.2%, MgO: 0.8%, Na <sub>2</sub> O: –, SO <sub>3</sub> : 0.2%	62 MPa (MWC + pre-curing)	–	[56]
Fly ash (Higher than 10% Ca component)	Porous morphology	MWC 200, 500, 700 and 850W MWC for 1 min	NaOH: 2, 5, 10 and 15 M Sodium Silicate (the weight ratio of Sodium silicate/NaOH was 2.5)	SiO <sub>2</sub> : 29.8%, Al <sub>2</sub> O <sub>3</sub> : 18.5%, Fe <sub>2</sub> O <sub>3</sub> : 16.8%, CaO: 19.8%, MgO: 1.9%, Na <sub>2</sub> O: 1.1%, SO <sub>3</sub> : 4.9%	–	0.9 g/cm <sup>3</sup> (MWC at 850 W with 15 M NaOH)	[39]

(continued)

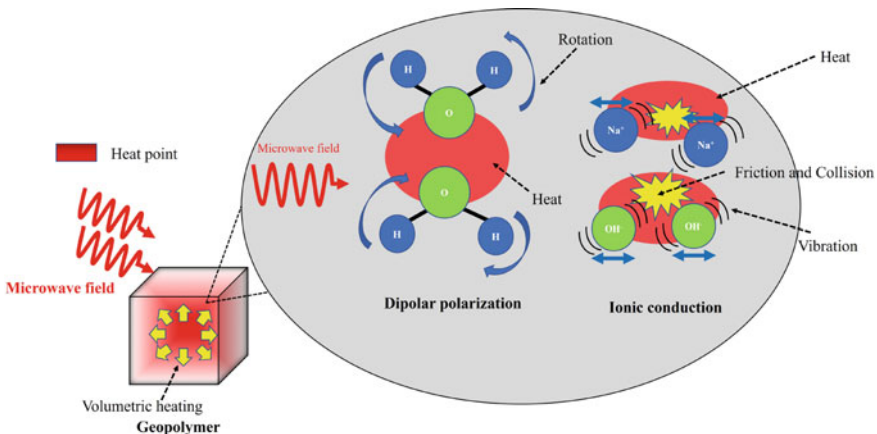


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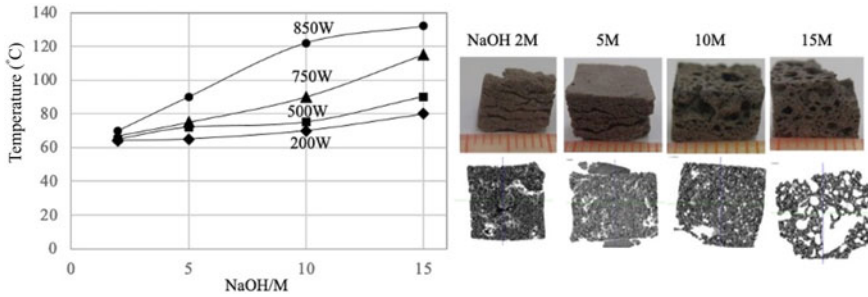
Raw materials	Morphology	Microwave curing (MWC) conditions	Geopolymerization conditions	Chemical composition (XRF)	Compressive strength (CS)	Density	Reference
Natural soil		MWC 575W MWC for 4 min + 1 min to eliminate the moisture	NaOH Na <sub>2</sub> SiO <sub>3</sub> (Alkali modulus of Na <sub>2</sub> O/SiO <sub>2</sub> was 1.25)	SiO <sub>2</sub> : 62.8%, Al <sub>2</sub> O <sub>3</sub> : 22.4%, Fe(OH) <sub>3</sub> : 11.5%, Calcite: 0.92%, MgO: -, Na <sub>2</sub> O: 0.8%, SO <sub>3</sub> : -	3.1 MPa (MWC with 50% natural soil)	0.44 g/cm <sup>3</sup> (MWC with 50% natural soil)	[16]
Bottom ash		MWC 900W MWC for 4 min	NaOH: 14 M Sodium Silicate	SiO <sub>2</sub> : 52.0%, Al <sub>2</sub> O <sub>3</sub> : 22%, Fe <sub>2</sub> O <sub>3</sub> : 17%, CaO: 6%, MgO: -, Na <sub>2</sub> O: -, SO <sub>3</sub> : -	3.4 MPa (MWC with 7% NaOH)	0.69 g/cm <sup>3</sup> (MWC at with 7% NaOH)	[57]

at 2.45 GHz, dipolar polarization generates heat in polar molecules such as water. Due to the electric fields associated with the microwaves the dipoles are aligned by rotation [45], providing more uniform heating [38, 45, 53]. In addition, in the high-alkali activator concentration, the access temperature was high [39], due to that in ionic conduction, the ions oscillate back and forth through the specimens by the electric field component of microwave irradiation. At the same time, heat generation by friction and collisions occurring between ions and molecules also supports heating [45, 53, 54]. Therefore, microwave devices are extremely convenient for influencing material reactions related to water at a frequency of 2.45 GHz, where the entire body quickly reaches a higher temperature by volumetric heating [41]. Because of microwave dielectric heating, water containing ions more efficiently heats in comparison to deionized water [55] due to ionic conduction and dipolar re-orientation variation with microwave frequency. In geopolymers,  $\text{Na}^+$  and  $\text{OH}^-$  ions play the ion conduction role, and heat is generated when the cations and anions collide with molecules and others (Fig. 2). By utilizing such advantages of microwave heating like instantaneous, very rapid heat-up time, and easy control, fast microwave synthesis of fly ash based porous morphology geopolymers was investigated in the presence of high-alkali concentration [39].

In the feasibility of synthesis of porous fly ash-based geopolymer using microwave energy, the geopolymer pastes were cured for approximately 1 min when a household microwave oven was operated at 200–850 W with high-alkali concentration. Figure 3 shows the temperature profile of geopolymer pastes containing different sodium hydroxide (NaOH) concentrations in different power of the household microwave for 1 min [39]. For the geopolymerization, NaOH solutions and sodium silicate solutions were used as activators, and microwaves oven was operated at a frequency of 2.45 GHz under identical conditions. The solid fraction in the geopolymer mixture



**Fig. 2** Illustration of microwave heating of geopolymer by ionic conduction and dipolar re-orientation variation



**Fig. 3** The plot of temperature against NaOH concentration of geopolymerized slurry solutions at different microwave oven powers (left), and pictures of geopolymers cured from components containing different NaOH concentration (upper right) and X-ray micro CT for the cross-section of geopolymers (lower right), where the microwave radiation was carried out at power 850 W for 1 min [39]

was 65 wt.%. The ratio of  $\text{Na}_2\text{SiO}_3/\text{NaOH}$  solution and  $\text{SiO}_2/\text{Al}_2\text{O}_3$  were fixed at 2.5 and 2.69, respectively, at NaOH solution concentration of 2–15 M.

Actually, as the ion concentration increased in the fly ash aqueous phase for geopolymers, heating occurred effectively by dipolar polarization. It was known about dielectric properties of geopolymer mortar constituents that fly ash, sodium silicate, NaOH and water had relative dielectric constants of 2.9, 2.78, 8.94, 14.32 and 72.18, and relative dielectric loss factor of 0.21, 0.20, 1.70, 1.17 and 11.75, respectively [58]. When NaOH concentration increased in the mortar, microwave heating easily occurs. In the results of relative dielectric constants and relative dielectric loss factor, water strongly contributes to the temperature increase in the slurry. In addition, at high NaOH concentration, the contribution influences the temperature increase. Therefore, as seen in Fig. 3 (left), high NaOH concentration enhances temperature increase. Also as shown in Fig. 3 (right) for the morphological observation of the resulting geopolymers, porosity developed in the matrix when the NaOH concentration became high. On the other hand, it is worth noting that geopolymerization is possible even in a low concentration NaOH environment. This is because microwave dielectric heating efficiently acts on geopolymerization at the molecular level. Namely, microwave generates a phase difference in the absorbed species between the orientation of the field and that of the dipole, causing energy to be lost from the dipole by molecular friction and collisions and thus heat, which leads to efficient geopolymerization [45, 46].

As the results show, the effect of microwave heating was summarized in the geopolymer structure with or without such porous morphology. The properties of geopolymer cured using microwave were explained with the aid of previous works.

First, as comparing the different properties of the porous and dense structure, in the cases of geopolymer of dense structure without micro-sized pores having the density of over  $1 \text{ g/cm}^3$ , reference [41] reported that compressive strength and bulk density of fly ash-based geopolymers were 12.9–42.5 MPa, and  $2.0\text{--}2.1 \text{ g/cm}^3$ . On the other hand, bottom ash-based geopolymers such as porous morphology geopolymer

obtained compressive strength and density of 3.6–6.2 MPa and 0.61–1.3 g/cm<sup>3</sup> [57]. Porous morphology geopolymer is very low compared to the compressive strength and bulk density of geopolymer using construction material [4, 10, 25, 26]. Therefore, porous morphology geopolymer is expected to be applied in lightweight concrete [44, 59] and adsorbent [8, 34] due to the low density.

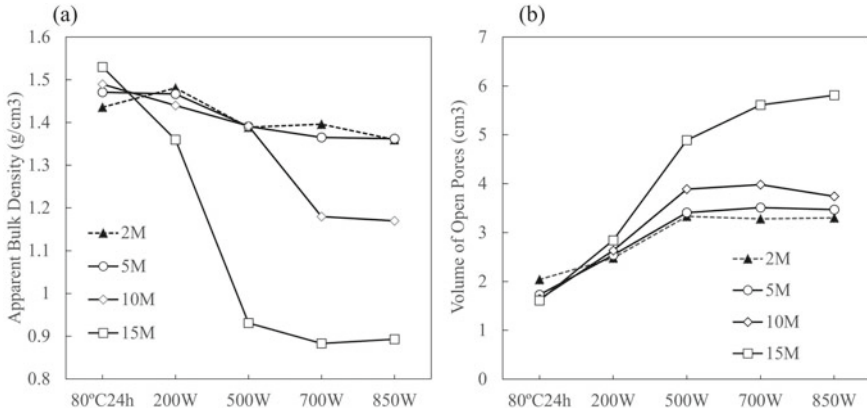
Second, the parameters affecting the properties of the geopolymer using microwave curing were microwave power, curing time, and concentration of alkali solutions. According to reference [42], the fly ash-based geopolymers were synthesized at different microwave powers of 200–600 W for 15 min. The compressive strength of geopolymers cured at 300 W increased (72 MPa), but at higher microwave power of 400 and 600 W it decreased (67 and 54 MPa). It was seen that higher microwave power led to surface cracks in the geopolymer bulk, because of highly evaporated water vapor. Thus, rapid dry curing is not useful for the high compressive strength of geopolymers at higher microwave power of 400 and 600 W. The compressive strength of geopolymer cured for different curing times of 10–60 min increased with increasing curing time, especially, geopolymer cured for 60 min recorded 90 MPa in short time. Reference [43] reported the effect of concentration of alkali solution on microwave curing. Fly ash-based geopolymers with NaOH concentration of 4–10 M were cured at 240 W for 120 min. The compressive strength of geopolymers increased with increasing NaOH concentrations. When NaOH concentration was 10 M, the compressive strength recorded the maximum value of 60 MPa. This is because increased dissolution rates of fly ash particles under higher NaOH concentrations may have formed higher amounts of alumino-silicate gel even at shorter curing time. Therefore, these factors affected the mechanical properties of geopolymers.

Finally, when comparing the effect of curing with and without microwaves, Chindaprasirt et al. [41] reported that fly ash-based geopolymers cured by oven heating at 65 °C for 24 h obtained compressive strength of 32.7 MPa and bulk density of 1.9 g/cm<sup>3</sup>, while geopolymers cured using microwave at power of 90 W for 5 min + additional oven heating at 65 °C for 12 h had higher compressive strength and bulk density of 42.5 MPa and 2.1 g/cm<sup>3</sup>. Therefore, microwave curing provided significantly reduced curing time shorter within only 5 min.

Consequently, microwave irradiation affects geopolymer properties of their structure in dense or porosity, depending on the microwave conditions of irradiation power and time. In addition, the resultant compressive strength of geopolymers had a tendency to increase when increasing microwave curing time. This is related to the longer cure times under microwave power, the increase of the internal temperature of the geopolymer matrix, and the promotion of geopolymerization [38, 42, 60]. However, much longer irradiation of microwave conversely and significantly affects the properties of the geopolymer, for example, decreasing the compressive strength [38]. As a result, the longer microwave treatment led to thermal shrinkage and provided thermal stress on the internal structure. Then, these reached the large cracks, pores, burns, and formed glassy bubbles in the geopolymer matrix [39, 61, 62]. Therefore, appropriate parameters need to be chosen for each geopolymer fabrication system by microwave curing.

### 3 Microwave Cured Geopolymer Having Water Foaming Agent

In the presented geopolymers, various properties were obtained depending on pore size in the cured bulk. When the pore size increased, the weight loss occurred, and surface area was increased in the case of high porosity structure [32, 33, 39, 63]. These geopolymers find applications for adsorbent [8, 34] and lightweight concrete [59], etc., where pore-forming agents like cooking oil and starch create voids within the mixture [64], surfactants [65], and  $H_2O_2$  when added to alkaline environment [66]. In the case of microwave heating to form the porous structure, the forming agent was water, and the porosity was caused by the modified temperature treatment, and the amount of water evaporation. Although it is difficult to control the microstructure of pore size, pore number, and pore distributions, there is an advantage in forming a porous matrix in the absence of any other foaming agents. Actually, the microstructure of the resulting geopolymers depends on the microwave condition with pre-curing as shown in Table 3. For the preparation of the geopolymer matrix, the SEM micrographs indicated a dense structure, having compressive strength in the range of 42–62 MPa [22, 41, 43, 56]. In these cases, it was convenient to operate microwave curing and conventional heating. In contrast, porous geopolymers were fabricated by microwave curing only, especially with high microwave power. This was due to microwave heating as water absorbed microwaves and generated vaporization as a result of polarization caused by external oscillation of polar water molecules [67, 68]. In the microwave curing, the water in the geopolymer paste was heated and then vaporized easily at temperatures higher than 100 °C, due to the generation of free water created bubble [68]. Then, the vaporization process may influence the morphology of the geopolymers, since bubbles caused by evaporation of water in geopolymer paste were fixed inside the geopolymer sample along with the heating curing process, resulting a porous morphology geopolymer structure [39]. Reference [39] also reported the investigation of the effect of the ratio of sodium silicate ( $Na_2SiO_3$ )/NaOH during the fabrication of fly ash-based geopolymer using the household microwave. When the water weight ratio in slurry paste increased, numerous pores formed in the geopolymer matrix, indicating that a lot of water evaporation occurred to cause pore voids during curing. In addition, the presence of NaOH in the geopolymer paste increased temperature, because dielectric heating was effective in higher NaOH. Thus, the microwave heating enhanced evaporation of water acting as a forming agent. This could be controlled to tailor the porosity and density of the geopolymers, depending upon microwave heating condition, but few studies on geopolymer foams using microwave heating. There were examples for microwave geopolymerization for porous fly ash-based geopolymer [40]. Although the fly ash-based geopolymers were cured by traditional oven heating at 80 °C for 24 h, they obtained an apparent bulk density of 1.5 g/cm<sup>3</sup>. The microwave cured geopolymers with 15 M-NaOH concentration decreased the apparent bulk density to 0.9 g/cm<sup>3</sup>. The effect of microwave powers and concentration of NaOH activator is investigated in Fig. 4a. In the higher microwave power with over 500–800 W, the



**Fig. 4** Apparent bulk density (a), and volume of open pores (b) of geopolymer cured at different microwave power 200–850W having different NaOH concentration and cured by oven at 80 °C for 24 h [39]

addition of NaOH concentration increased the volume of pores in the geopolymers (Fig. 4b) [39].

Actually, this section summarizes microwave curing for geopolymer to the effective to porous forming without forming agents. Especially, higher microwave power promoted to the more porous structure in the geopolymer.

#### 4 Pre-curing and Microwave Cured Geopolymer Concretes

The most practical application of geopolymers is as a replacement material for existing concrete [9, 69–72]. This is because of the fact that cement manufacture is highly energy intensive and emits CO<sub>2</sub> and other greenhouse gases responsible for global warming [69, 73]. In this way, the use of ceramics as structural materials requires a dense structure and high mechanical properties. In these cases, the geopolymer is densely packed, providing less pores. But, the microstructures of geopolymer mortars were influenced by the use of conditions under different temperatures [74], crystalline compound [75], alkali activator [76, 77], and silica to alumina ratio in the geopolymer components [78, 79].

Due to dipolar polarization and ionic conduction mechanisms, microwave heating was effective for geopolymerization in the advantage of short time preparation. If anything is ideal to decrease the evaporation of water during microwave heating, resulting in less evaporation of water even at over 100 °C, microwave radiation effectively enhanced the geopolymerization. Actually, microwave heating led to high compressive strength of fly ash geopolymers [40, 41]. But, common geopolymerization can take a long curing period to prepare the bulk ceramic body from a few days to as long as 90 days in geopolymer mortars [78, 80]. But, if pre-curing and

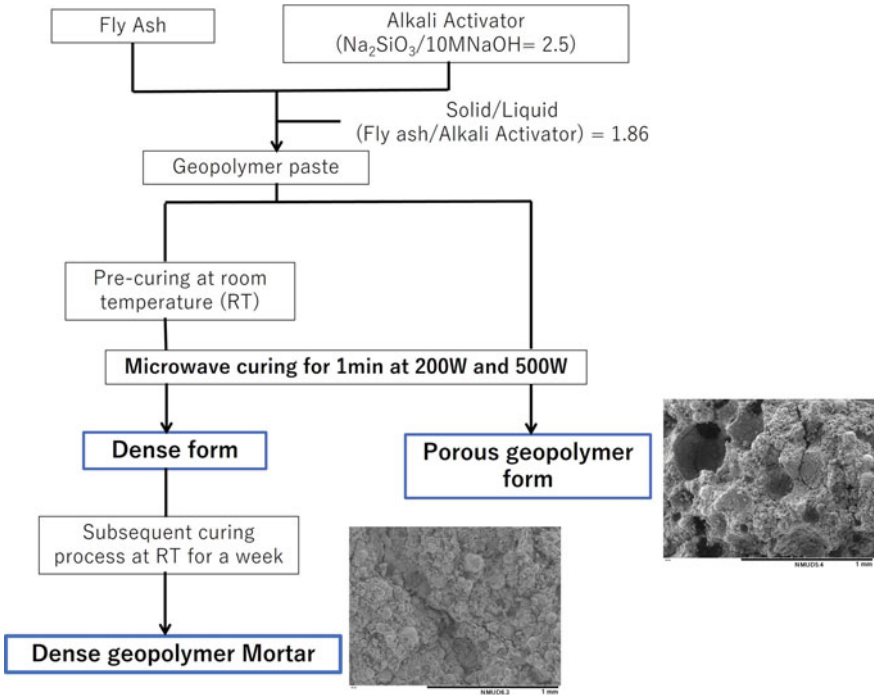
microwaved curing were combined, the microwave method diversified to prepare densely structured geopolymers (Fig. 5). In the case of microwave heating, the same formulation composition can be changed into porous morphology geopolymer and dense structure geopolymer without micro-sized pores by changing the adjustment conditions. In the geopolymer paste made from fly ash [39], microwave heating can easily produce porous morphology geopolymer without pore forming agents. For example, when the geopolymer paste was formed with the weight ratio of (Solid/Liquid) = 1.86 and ( $\text{Na}_2\text{SiO}_3/10\text{ M-NaOH}$ ) = 2.5, porous morphology geopolymer was produced within 1 min by simply exposing microwave as shown in the SEM picture inserted in Fig. 5. On the other hand, pre-curing at room temperature after preparing the geopolymer paste could prepare the geopolymer with dense structure. Actually, after the microwave curing for 1 min at both 200 W and 500 W out-put power, the value of the compressive strength (MPa) without and with a 1 h pre-curing process at room temperature (RT) increased from 5.8 MPa to 6.2 MPa, and 1.5 MPa to 3.9 MPa in the case of the 200 W and 500 W of microwave out-put power respectively as shown in Fig. 6. When the geopolymer was without pre-curing, compressive strength decreased due to the formation of pores. This means that pre-curing was possible to easily control the fabrication of the geopolymer dense structure without micro-sized pores with high compressive strength. In addition, the compressive strength of geopolymers cured by conventional oven curing at 25 °C for 7 days was 4.4 MPa, whereas geopolymer with 1 h pre-curing and microwave cured at 200 W for only 1 min was 6.2 MPa.

So, in summary, this section summarized the effect of microwave curing and pre-curing for densely structured geopolymers. By combining both curing methods, it would be possible to have the practical application of the resulting geopolymers, because of shortening the preparation period.

## 5 Future Prospects of Geopolymers

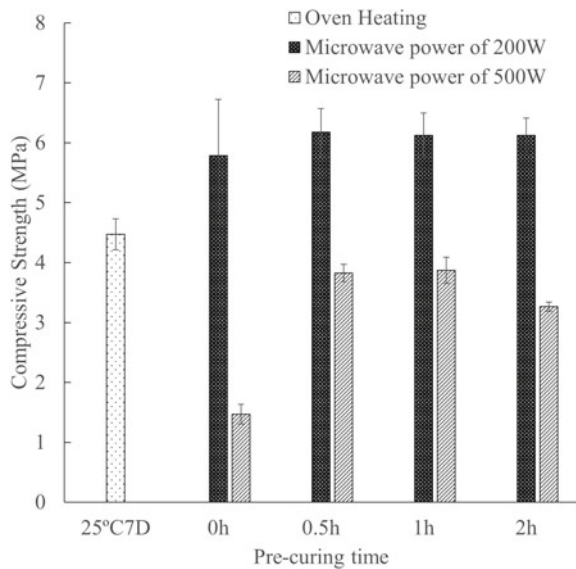
Given the environmental footprint of geopolymer production and operation, the benefits of concrete mainly lie in the sustainability of geopolymer production with the manufacturing process including lower resource and energy requirements and lower CO<sub>2</sub> emissions [23, 81, 82]. In addition, there are other benefits of geopolymers such as reuses of industrial waste and production of much less fuel, which leads to contribute in the reduction of global warming. From this point of view, geopolymers can fully play the role of alternative concrete. Recently world's first building with geopolymer concrete has been constructed at the University of Queensland's Global Change Institute and also at the Brisbane West Well camp airport in 2014, as applied by the world's largest geopolymer concrete project [82].

As for the future prospects of geopolymers, their industrial applications are of great interest. Many review articles recently summarized these applications [21, 66, 69, 71, 72]. To summarize in terms of geopolymer material form, we can consider bulk body, form body, composite, and coating as shown in Table 4. Among their



**Fig. 5** Flowchart of microwave heating for preparation of porous and dense geopolymers sourced from fly ash and sodium silicate (Na<sub>2</sub>SiO<sub>3</sub>) in NaOH activator

**Fig. 6** Compressive strength of geopolymer without pre-curing and with various pre-curing periods of time. Geopolymers were synthesized at microwave power 200 and 500 W, cured for 1 min, and at 25 °C for 7 days



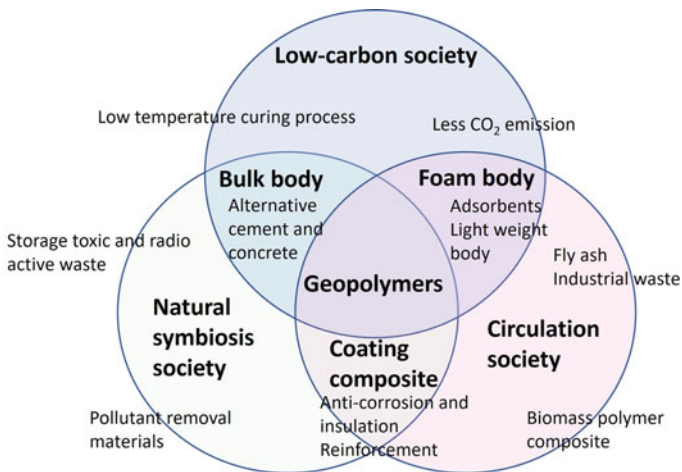


applications, the most common research case is the application of concrete, mortar, and cement for building materials. The application fields of these geopolymers have the potential to contribute significantly to the construction of a sustainable society (Fig. 7).

In particular, apart from the sustainability aspects of the geopolymer binder, the synthesis conditions become important factors affecting the environmental footprint

**Table 4** Applications of geopolymer materials having different forms

Form of geopolymer material	Applications	References
Bulk body	Cement and concrete	[4, 25, 26, 29, 47, 69, 71]
	Ceramics	[83–85]
	Storage toxic, and radioactive waste	[1, 86–88]
Foam body	Adsorption of heavy metal	[8, 34]
	Light weight cement	[44, 59]
Coating	Anti-corrosion and acid and base resistant	[80, 89–91]
	Reinforcing	[92]
	Temperature resistance	[89, 93]
Composite	Fire and heat resistant matrix materials	[44, 81, 94]
	Reinforcing	[95, 96]



**Fig. 7** Geopolymers eco-friendly cooperated with the integrated improvement of economy and environment for a sustainable society having symbiosis with human and nature. Low carbon, circulation and natural symbiosis are reached by environmental life and civilization under SDGs-ship [97]

of the production. In addition, immobilization and adsorption of pollutant metals are interesting technologies of geopolymers using bulk and foam geopolymers. Geopolymer layer coating technology on the substrates could also show reinforcement of the substrate materials and provide insulation against acid or base attack and corrosion of the body materials. Similar effects were found in geopolymer composite with organic polymers and other additives. In the future when dealing with these technologies of geopolymers, it is necessary to consider the concept containing a low-carbon society, circulation society, and natural symbiosis society. As discussed before, if geopolymer is synthesized by a common sintering process, the environmental footprint of the product may be not comparable to concrete materials, due to the consumption of high energy associated with sintering, becoming less “green” process. In other words, the low carbon and low power consumption of geopolymer processing are attractive and consistent with the goals of a sustainable society [98].

## 6 Final Remarks

This chapter highlights geopolymers prepared by microwave treatments, specifically how their properties can be tuned using microwaves. The incorporation of microwave heating in the geopolymer production process has the advantages of being able to adjust the porous morphology geopolymer without additives, and to make dense structure geopolymer without micro-sized pores concreted under certain conditional settings. Due to the incorporation of microwave heating in increasing efficiency and improving characteristics with less energy consumption, it is expected to be used as an eco-friendly ceramic material in the future. However, several problems such as difficulties in temperature control and the low energy conversion efficiency remain in the practical application of microwave cured geopolymers. Although this method is still in a transitional stage, it has features not found in conventional methods, and thus holds promise for the future.

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