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Preparation and properties of metakaolin-based porous geopolymer formed with sodium perborate

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ABSTRACT

This work aimed to study the use of sodium perborate as a foaming agent in the production of porous metakaolin-based geopolymer. The influence of sodium perborate foaming agent on physical properties, mechanical properties, and thermal conductivity was focused. The results revealed that the porosity and pore size increased with an increment of the additions of sodium perborate from 0.5 to 2.0 wt.%. The obtained porous geopolymers showed compressive strength of 5–6 MPa and thermal conductivity in the range of 0.22–0.32 W/mK. The addition of sodium perborate produced porous geopolymer with acceptable compressive strength and thermal conductivity.

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KEYWORDS

Geopolymer; sodium perborate; mechanical strength; porosity; thermal conductivity

1. Introduction

Geopolymer is a new amorphous to semi-crystalline inorganic material that is synthesized by the reaction of rich alumina and silica sources with alkaline solution at ambient temperature to temperatures lower than 100°C [1–3]. The aluminosilicate framework of geopolymer leads to excellent properties such as high strength, chemical resistance, and fire resistance. Because of these properties, geopolymer becomes an alternative material for building materials. In recent years, there has been a significant increase in the demand of lightweight building materials with a good thermal insulating property [4]. Compared with conventional thermal insulation materials, geopolymer has received a lot of attention due to its low energy process.

Several methods have been developed to produce porous geopolymers [5–7] such as lightweight fillers addition [8,9], direct foaming method [10], replica method [11], additive manufacturing [12,13], water-soluble pore-foaming agent addition [14], and the mixing of several methods [15]. Direct foaming method has been widely studied because of its simplicity [5–7,10]. The commonly used foaming agent are aluminum (Al), silicon (Si), and hydrogen peroxide (H_2O_2) [16–20]. Since the geopolymer foamed using H_2O_2 showed finer pore distribution than that using Al [21], H_2O_2 has got a lot of interests [22–28]. However, it can dissolve because of light. Therefore, H_2O_2 must be stored in cool place and its high concentration cannot directly contact [29].

Sodium perborate (NaH_2BO_4) is a chemical salt, which is generally used in the laundry detergent and cleaning products. NaH_2BO_4 is more stable and easier to handle than H_2O_2 . It can be used for the same purpose as H_2O_2 [29,30], and it has been recently reported as a new effective foaming agent for producing porous geopolymers because the use of NaH_2BO_4 foaming agent showed lower cost to thermal resistance ratio than the use of H_2O_2 [31]. Thus, NaH_2BO_4 should be a choice for foaming agent in the manufacturing of porous geopolymer.

In addition, the preparation of porous geopolymer by direct foaming with sodium perborate (NaH_2BO_4) was very rarely reported in the literature. Therefore, the utilization of sodium perborate as foaming agent in metakaolin-based geopolymer is studied in this work, and the results is reported in this paper.

2. Materials and methods

2.1. Raw materials

Metakolin was received by calcining kaolin clay from Ranong province in the southern part of Thailand at 600°C for 2 h. The conversion of kaolin clay to metakaolin via dehydroxylation reaction was confirmed by XRD analysis as shown in Figure 1. Its chemical composition is presented in Table 1, which shows the content of SiO_2 and Al_2O_3 is about 95%. Sodium perborate (NaH_2BO_4) supplied from Elago Enterprise Pty Ltd. was used as the foaming agent.

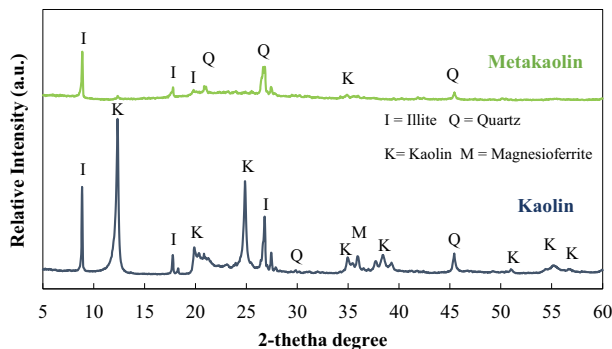


Figure 1. XRD patterns of kaolin and metakaolin.

Table 1. Chemical compositions of metakaolin.

Composition	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	K ₂ O	Na ₂ O	CaO	MgO	Others
Metakaolin	54.48	41.30	1.29	2.40	0.05	0.02	0.07	0.39

2.2. Porous geopolymer preparation

The alkaline activator was prepared by mixing 10 M sodium hydroxide (NaOH) solution with sodium silicate (Na₂SiO₃) solution at the weight ratio of 2:3. First, the metakaolin was blended with sodium perborate (NaH₂BO₄) by stirring at a speed of 750 rpm for 1 min and then mixed with the alkaline activator solution for further 5 min to form geopolymer paste. In this study, the weight ratio of solid:liquid content of 55:45 was employed. The amount of sodium perborate foaming agent varied from 0.5 to 2.0 wt% of metakaolin. Geopolymer paste was poured into 25 mm × 25 mm × 25 mm silicone molds for cube shape and 25 mm × 25 mm × 120 mm silicone molds for bar shape. After demolding, the geopolymer samples were sealed with plastic film and cured at 27, 40, 50, and 60°C for 24 h. Afterward, the samples were wrapped with plastic sheet and aged at 27°C and 75% relative humidity until the required ages (28 days).

2.3. Characterization

Phases of porous geopolymer was analyzed by using an x-ray diffractometer (XRD, D8 Advance, Bruker). Physical properties i.e. water absorption, bulk density, and apparent porosity were carried out by using an Archimedes method according to ASTM C373. The total porosity was calculated following the Equation (1).

$$\text{Total porosity}(\%) = \left[1 - \frac{\text{bulk density}}{\text{true density}} \right] \times 100 \quad (1)$$

Compressive strength and three-point bending strength (flexural strength) were tested by using a universal testing machine (Instron 8872) at a crosshead speed of 1 mm/min. Pore morphology was observed by optical microscope (Stereo microscope, Stemi-2000). Software Image J program was

employed to analyze the pore size distribution by using the images with at least 100 pores per each sample. Thermal conductivity was examined by Thermal Conductivity Analyzer (TCA, model Hot Disk TPS 2500 S) using the transient plane method with a hot-disk sensor. To ensure accuracy, each sample was measured three times at room temperature and the average value was reported.

3. Results and discussion

3.1. Effect of foaming agent (NaH₂BO₄) on physical properties

Figure 2 shows bulk density and water absorption of the geopolymer with the different amount of foaming agent addition. Bulk density of geopolymer without the addition of foaming agent was 1.43 g/cm³ (1429 kg/m³) and decreased to 1.08 g/cm³ (1077 kg/m³) when 0.5 wt% of foaming agent was added. The lowest bulk density was found in the geopolymer with the addition of 2 wt% foaming agent. Water adsorption of porous geopolymers increased with an increase in the amount of foaming agent.

Total porosity of the geopolymer with the addition of various amount of foaming agent is shown in Figure 3. Total porosity of geopolymer increased as the amount of foaming agent increased. The addition of 0.5 wt% Na-perborate (NaH₂BO₄) foaming agent resulted in a significant increase of porosity, and the total porosity was gradually increased when further increasing amount of foaming agent was added.

3.2. Effect of curing temperature

Figure 4 illustrates total porosity of porous geopolymer cured at different temperatures. It is clearly seen from this figure that the effect of curing temperature on the formation of the pore was not found. This indicated that the temperature at 27°C was enough to allow the

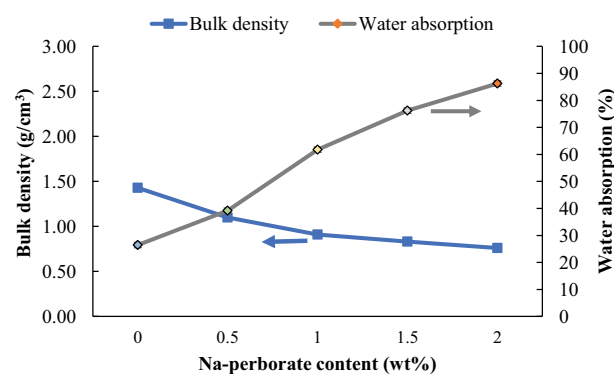


Figure 2. Bulk density and water absorption of the geopolymer with the different amount of foaming agent addition.

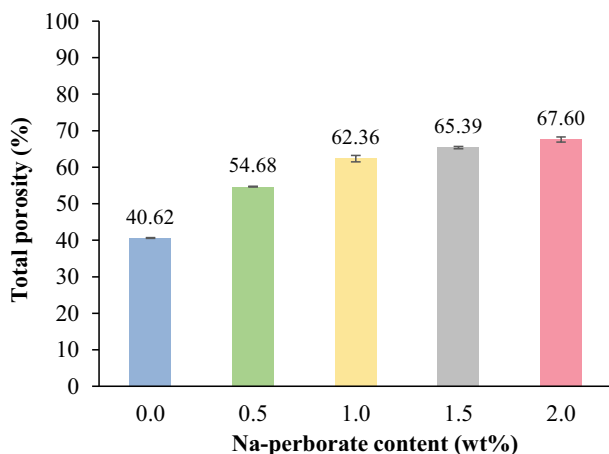


Figure 3. Total porosity of the geopolymer with the addition of various amount of foaming agent.

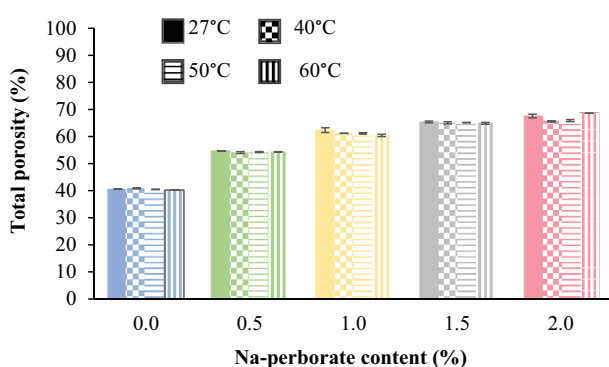


Figure 4. Total porosity of porous geopolymer cured at different temperatures.

reaction of sodium perborate with water to form the pores. Therefore, the curing temperature at 27°C was selected for further experiment.

3.3. Phase analysis and pore morphology

XRD patterns of the geopolymer with the addition of various amount of foaming agent as shown in Figure 5 demonstrate the presence of amorphous phase of aluminosilicate together with unreacted phase of

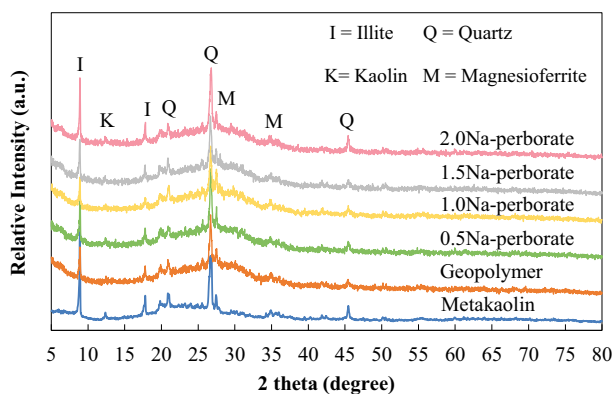
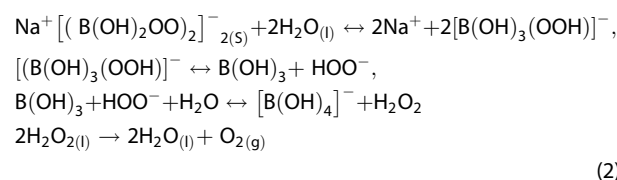


Figure 5. XRD patterns of the metakaolin and geopolymer with the addition of various amount of foaming agent.

metakaolin such as kaolin, illite, quartz, and magnesioferrite in all geopolymers. No difference of phases was detected in the porous geopolymer with the addition of foaming agent.

Figure 6 presents the pore morphology of porous geopolymer with the addition of various amount of foaming agent. The geopolymer without the addition of foaming agent showed the dense geopolymer matrix with a few pores, while many pores with various sizes were observed in the porous geopolymer with the addition of foaming agent. This result implied that the addition of NaH_2BO_4 foaming agent influenced the pore characteristic. The amount of pore increased with an increase in foaming agent content. Sodium perborate (NaH_2BO_4) was dissolved in the water and reacted with water to form oxygen gas as given in equation (2) [32]. These oxygen gases generated the pores in geopolymer matrix similar to H_2O_2 [28].



In addition, the tiny spherical pores coalesced when a higher content of foaming agent was added. An increment of the foaming agent content enhanced the formation of larger size of pores in the level of macropores as shown in Figure 7. This was due to thermodynamically unstable process of the foaming agent. The gas bubbles from its decomposition within the paste coalesced and expanded to become macropores [28,33]. In order to solve this problem, the stabilizing agent was added [33–35]. Since different types of stabilizing agent showed different effect on the foam and geopolymer paste [22–28,33–35], more investigation on stabilizing agent and its parameter process will be conducted to further explain their behaviors.

Despite being thermodynamically unstable, which caused inhomogeneity of the pores, it has been reported that an advantage of NaH_2BO_4 was the slower decomposition rate, which is capable to control the beginning of the foaming process [31,36].

During the decomposition process of NaH_2BO_4 (equation 2), boron in the form of borate is formed, which may pose a risk to the user's health because borate is classified as a CMR substance in category 1B of Commission Regulation (EC) 790/2009, leading to the use of borates being forbidden in cosmetics [37]. Nevertheless, some borates including NaH_2BO_4 still have been used in other products such as detergents, bleaches, and disinfectants by following the safety terms and conditions [38]. In addition, geopolymer has been ordinarily recognized to use for immobilizing the toxic wastes [36,39]. Immobilization of boron and borate salt was also investigated by several researchers. The study by Palomo and López de la Fuente found

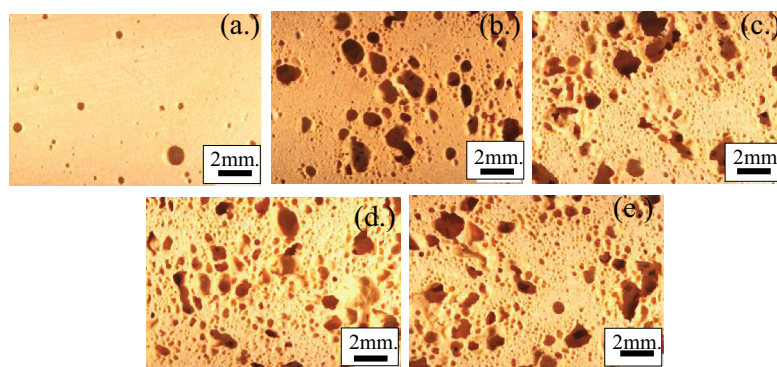


Figure 6. Optical microscope geopolymer and porous geopolymers: (a) geopolymer, (b) porous geopolymer with 0.5% Na-perborate, (c) porous geopolymer with 1.0% Na-perborate, (d) porous geopolymer with 1.5% Na-perborate, and (e) porous geopolymer with 2.0 % Naperborate.

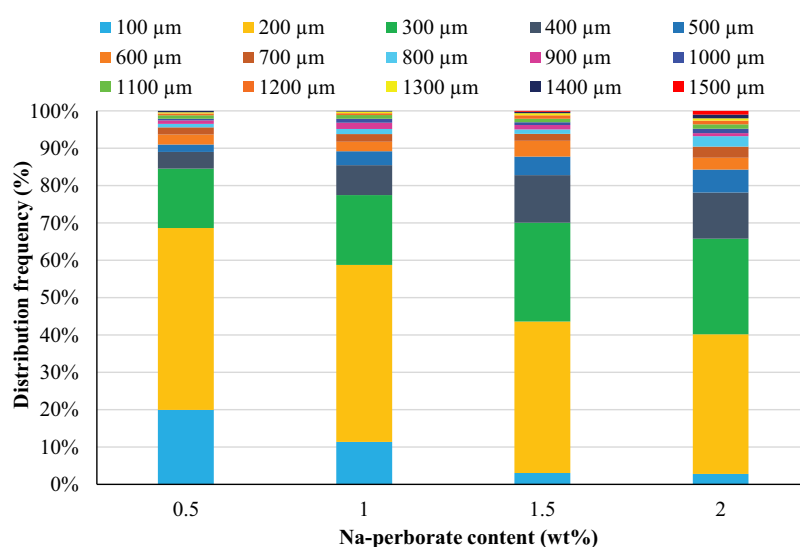


Figure 7. Pore size distribution of porous geopolymer with the addition of various amount of foaming agent.

that boron was effectively immobilized in geopolymer [40]. Taviri et al. have carried out research using solid-state MAS NMR spectroscopy. From their results, they concluded that boron was a part of the geopolymer structure. It played a role in the network of geopolymer structure like aluminum [41], which was difficult to release. Therefore, the obtained porous geopolymer foamed with NaH_2BO_4 might be safe for use in the products. However, further experiment on the leaching of boron will have to be performed to ensure this point.

3.4. Mechanical properties of porous geopolymer

The porous geopolymer exhibited a decrease of compressive strength with an increment of foaming agent content as demonstrated in Figure 8(a). The compressive strength of the geopolymer without any addition was around 51–62 MPa. Addition of 0.5wt% of NaH_2BO_4 foaming agent drastically reduced the compressive

strength, and the compressive strengths were steady as further increasing foaming agent contents were added. The compressive strength of the obtained porous geopolymer was in the range between 5 and 6 MPa. This compressive strength value was higher than those reported of 4.8 MPa by Phavongkham et al. [42], who examined the compressive strength of fly ash-based geopolymer foam made with NaH_2BO_4 and surfactant.

A comparison of compressive strength of the obtained porous geopolymer with other porous geopolymers is given in Table 2. As shown in Table 2, very low compressive strength was generally obtained in porous geopolymers. For instance, Jaya et al. reported the compressive strength of 0.4–6 MPa for metakaolin geopolymer foamed with H_2O_2 and polyethylene glycol (Tween 80) [43]. The compressive strength of 3.34 MPa was achieved by Samson et al. [44] for H_2O_2 and surfactant-foamed metakaolin geopolymer. Le et al. obtained the compressive strength of 1.94–9 MPa for metakaolin

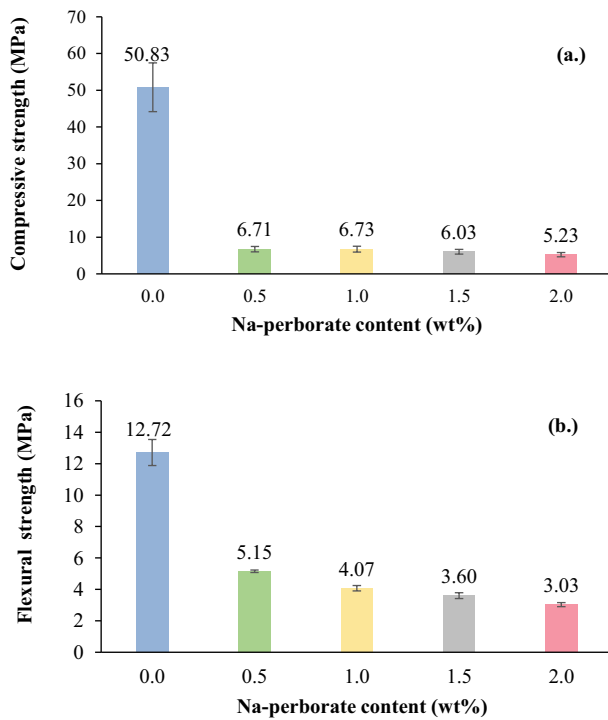


Figure 8. The compressive strength (a) and flexural strength (b) of geopolymer with the addition of various amount of foaming agent.

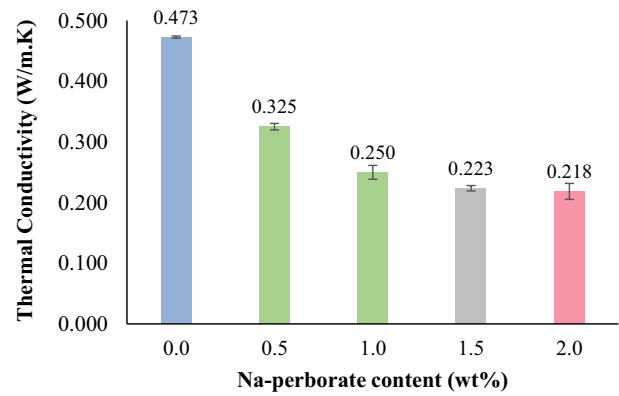


Figure 9. Thermal conductivity of geopolymer and porous geopolymers.

3.5. Thermal conductivity

Thermal conductivity of geopolymer and porous geopolymers is shown in Figure 10. Compared with the geopolymer (0.47 W/mK), the thermal conductivity of porous geopolymer reduced to about 0.325–0.218 W/mK. The thermal conductivity of porous geopolymers decreased with increasing of the foaming agent. These thermal conductivity values were comparable with

Table 2. Compressive strength of metakaolin-based porous geopolymer.

Matrix	Foaming agent	Compressive strength (MPa)	Ref.
MK	NaH ₂ BO ₄	5–6	This work
MK	H ₂ O ₂ + polyethylene glycol (Tween 80)	0.4–6	[43]
MK	H ₂ O ₂ + commercial surfactant	3.34	[44]
MK	Al	1.94–9	[45]
MK	H ₂ O ₂ + sodium dodecyl sulfate	0.1–5.7	[46]
MK	Silica fume + rice husk ash, sand, sawdust	1–4	[47]
MK	H ₂ O ₂ + olive oil	0.2–3.9	[22]
MK	H ₂ O ₂ + canola oil	0.3–5.7	[23]
MK	H ₂ O ₂ + cetyl trimethyl ammonium bromide	0.3–5.9	[28]
MK, calcined clay	H ₂ O ₂ + butter, pork lard	0.6–2.5	[48]
MK, soda lime	H ₂ O ₂ + polyoxyethylene octyl phenyl ether (Triton X-100)	0.4–5.5	[49]
MK, FA	H ₂ O ₂ + calcium stearate	2.1–5.4	[50]

geopolymer foamed with Al powder [45]. From Le et al.'s study, the high compressive strength was probably due to the fiber reinforcement.

Similarly, the downward trend with the addition of foaming agent was observed for flexural strength (Figure 9(b)). Among the porous geopolymer, the maximum flexural strength of 5 MPa was achieved in porous geopolymer containing 0.5 wt% NaH₂BO₄, and the flexural strength gradually decreased with an increase in foaming agent.

The above results suggested that the strength was correlated with the density, and the reduction of the strength was due to the increment of pores developed in the porous geopolymer.

According to TIS 58–2533 standard, the minimum limit for compressive strength of conventional non-load-bearing concrete masonry is 2.5 MPa [51]. The compressive strength of the obtained porous geopolymer was higher than that indicated in the standard.

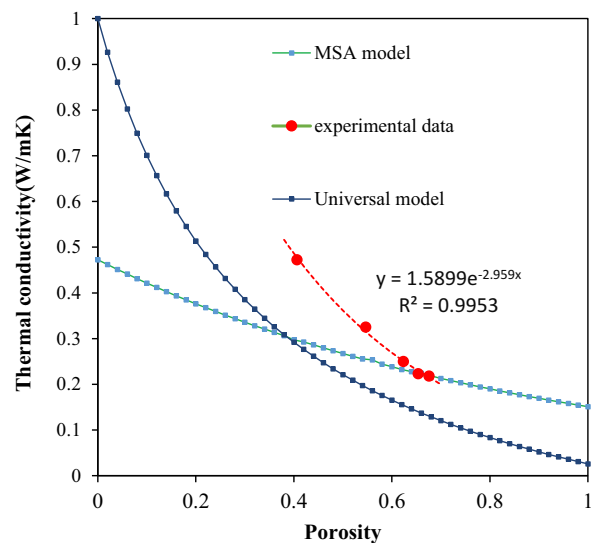


Figure 10. The correlation obtained following the minimum solid area (MSA) and the universal model.

Table 3. Properties of lightweight concrete.

Property	Materials				
	Lightweight concrete			Nonload-bearing concrete masonry [51]	Porous geopolymer (this work)
	Class I structural	Class II structural/insulating	Class III insulating		
Compressive strength (MPa)	>15	>3.5	>0.5	>2.5	5–6
Thermal conductivity (W/mK)	-	<0.7	<0.3	0.436–0.456	0.218–0.325
Density (kg/m ³)	1600–2000	<1600	<<1450	-	760–1077

those reported by Phavongkham et al. (0.27–0.32 W/mK) for fly ash geopolymer foamed with NaH₂BO₄ and surfactant [42], by Pantongsuk et al. (0.15–0.45 W/mK) for H₂O₂-foamed metakaolin-blended bagasse ash geopolymer [52], and by Le et al. (0.15–0.36 W/mK) for metakaolin geopolymer foamed with Al powder [45].

The thermal conductivity values of various types of lightweight concrete and nonload-bearing concrete masonry are summarized in Table 3. The obtained thermal conductivity values of porous geopolymer in this study were lower than that of conventional nonload-bearing concrete masonry. The values of thermal conductivity of the obtained porous geopolymer were in the range of Class II lightweight concrete [53,54].

In accordance with Table 3, the obtained porous geopolymer was a good candidate for using as structural and thermal insulating lightweight materials as well as lightweight concrete.

There are several models proposed to describe the correlation between thermal conductivity and porosity. Among these models, the modified minimum solid area (MSA) model proposed by Rice [55–57] and the universal model [58,59] were widely used. The modified equation of MSA was given in equation (3), and the universal model was expressed by equation (4).

$$\lambda = \lambda_0 \exp(-b^2 \epsilon) \quad (3)$$

where λ_0 is the thermal conductivity of dense geopolymer and b is an empirical constant.

$$\lambda = \frac{\sum_{i=1}^m \lambda_i V_i ((d_i k') / (d_i - 1) k' + \lambda_i)}{\sum_{i=1}^m V_i ((d_i k') / (d_i - 1) k' + \lambda_i)} \lambda \quad (4)$$

where λ and V are thermal conductivity and porosity, m is number of component, and i represents the component. d_i is a parameter related with pore shape and k' is a parameter that reflected the heat conduction between solid and air.

The relationship between thermal conductivity and porosity of porous geopolymers is shown in Figure 10. The experiment data were rather close to those of the universal model than MSA model. Although some deviation of the experiment data from the universal model was found, the R^2 of 0.996 was obtained. This high correlation factor value suggested that total porosity

was an important parameter which influenced the thermal conductivity. The deviation of experiment data is attributed to the effect of the distribution of pore, pore shape, and pore connectivity [60].

4. Conclusion

This work studied mainly the preparation and properties of porous metakaolin-based geopolymers foamed with sodium perborate (Na-perborate) foaming agent. The result showed that Na-perborate can be utilized as a foaming agent in geopolymer as well as H₂O₂. The presence of Na-perborate increased the porosity while decreased the bulk density, compressive strength, flexural strength, and thermal conductivity. All porous geopolymers exhibited flexural strength in the range of 3.0–5.2 MPa. The optimum compressive strength of 5 MPa and thermal conductivity of 0.2 W/mK were obtained in the porous geopolymer with the addition of 2 wt% Na-perborate. The obtained porous geopolymers has a potential to be used as a structural and thermal insulating material in building and construction industries.

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Disclosure statement

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