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Dielectric properties and temperature profile of fly ash-based geopolymer mortar ☆

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ABSTRACT

In this paper, the fresh properties, dielectric properties and temperature profile of fly ash-based geopolymer mortars are investigated to observe the effect of mixture proportions and time after mixing. Sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3) were used as the activators. The results of fresh properties revealed that the added water could improve the workability of geopolymer mortar. It is also found that the initial and final setting times of all the geopolymer mortars are less than those of cement mortars. For the dielectric properties and surface temperature, mortars were measured during a 24-hours geopolymerisation period at room temperature. The obtained results show that dielectric properties and surface temperature of geopolymer mortars tended to decrease with increasing time after mixing. And the geopolymer mortars had the highest value of dielectric properties and surface temperature at right after mixing. In addition, the dielectric properties (ε_r' and ε_r'') of the specimens with more liquid constituents were also higher. In particular, this work shows that it is possible to cure the fly ash-based geopolymer mortars with microwave energy depending on the mixture proportion and time after mixing.

1. Introduction

The term 'geopolymers' was first introduced by Davidovits in 1979 to designate a new class of three-dimensional alumino-slilicate materials [1]. Geopolymer is the new cementitious binder material. The synthesis of geopolymer takes place by polycondensation from the reaction of a solid aluminosilicate with a highly concentrated aqueous alkali hydroxide or silicate solution. Geopolymeric materials are attractive because excellent mechanical properties and durability can be achieved [2,3]. The wide variety of potential applications of geopolymer-based materials includes: new ceramics, cements, matrices for hazardous waste stabilization, fire-resistant materials, asbestos-free materials and high-tech materials are some of the potential uses of geopolymers [4–8]. Normally, geopolymer is mixed at room temperature and then cured in ranking from room temperature to 95 °C for about 6 h to 4 days [9]. The conventional heating methods to cure geopolymer require heat conduction from material's outward surface and these methods are slow and inefficient for materials that poorly conduct heat.

Unlike the conventional heating, microwave is an attractive method to heat materials. Microwave generates heat within the material and heats the entire volume at about the same rate. By the microwave heating differs by means of the in-situ method of heating providing the prospect of uniform temperature distribution, improved heating efficiencies and was pollution-free environment since there are no products of combustion [10]. In contrast to that in conventional drying, microwave drying gives higher temperatures inside the drying sample while the surface temperature stays colder due to the cooling effect of ambient air [11]. The principles and advantages of microwave heating suggest that microwave energy can be applied for geopolymer curing. Knowledge of the dielectric properties is useful in studying and developing heating processes or grading techniques based on electromagnetic energy since electric conduction, dipoles, electronic, ionic and Maxwell–Wagner mechanisms directly influence materials' dielectric properties and are closely dependent on electromagnetic wave frequency [12]. Therefore, in order to confirm the possibility of using microwave heating in geopolymer, dielectric properties of materials must be examined to verify the curing. The ability of a dielectric to be polarized is expressed in terms of the 'electric permittivity' of the material. Most materials exhibit a difference in phase, in which the polarization movement lags behind the alternating electric field frequency and indicates a loss in conductivity within the material filling the cavity. To account for this change in conductivity, the electric permittivity is described as a complex quantity (Eq. (1)) with both real and imaginary parts (the imaginary part accounts for the loss in conductivity in the material) [10]:

$$\epsilon^* = \epsilon' - j\varepsilon'' \tag{1}$$

ϵ': real permittivity (F/m), ε'': imaginary permittivity (F/m), ϵ^*: complex permittivity (F/m), j: $\sqrt{-1}$ The relationship between the real

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Nomenclature

$\tan\delta$	Loss tangent
ε'	Real permittivity (dielectric constant) (F/m)
ε''	Imaginary permittivity (loss factor) (F/m)
ε^*	Complex permittivity (F/m)
ε_r'	Relative dielectric constant
ε_r''	Relative dielectric loss factor

and imaginary parts is expressed as the loss tangent (Eq. (2)). The loss tangent ($\tan\delta$) can be used to represent the fraction of stored energy lost per period of field oscillation:

$$\tan\delta = \varepsilon_r'' / \varepsilon_r' \quad (2)$$

The objectives of this research are to study the influences of both the time after mixing and mix proportions to the fresh and the dielectric properties of fly ash-based geopolymer during a 24-hours geopolymerisation period. In addition, its surface temperature is observed since it is well known that complex dielectric properties are temperature dependent.

2. Materials and methods

In this section, the details of material, mixture proportion and mixing and casting to prepare the geopolymer mortar are shown.

2.1. Materials

The constituents of geopolymer mortar were fly ash, sodium hydroxide (NaOH) solution, sodium silicate (Na_2SiO_3) solution, sand and tap water. Lignite fly ash (FA) from Mae Moh power station in Thailand was used as a source material. The chemical composition of fly ash was 36.4% SiO_2, 20.5% Al_2O_3, 15.8% Fe_2O_3, 16.4% CaO, 3.6% MgO, 2.3% K_2O, 0.9% Na_2O, 3.4% SO_3, 0% TiO_2 and 1.2% Free CaO. The specific surface area (Blaine) of fly ash was 2133 cm^2/g. Sodium hydroxide solutions of 10 M concentration were prepared by dissolving NaOH pellets in tap water. Sodium silicate solution with composition of 28.75 SiO_2 wt.%, 9.58 Na_2O wt.%, and 61.67 wt.% water was also used. River sand passed sieve No. 16 (1.18 mm opening) and retained on No. 100 sieve (150 μm opening) with fineness modulus of 2.8 and specific gravity of 2.65 was used.

2.2. Mixture proportions

Nine mixtures of geopolymer mortar were prepared. The parameters of geopolymer mortar are as follows:

1) The weight ratios of fly ash-to-alkaline solution (FA/AS) were 2.0 and 2.5
2) The weight ratios of sodium silicate-to-sodium hydroxide solution (NS/NH) were 0.5, 1.0 and 2.0.

All geopolymer mortars were made with the weight ratios of 2.0 sand-to-fly ash.

2.3. Mixing and casting

The fly ash and NaOH solution are firstly mixed together in a Hobart mixer for about 5 min with the speed paddle of 285 ± 10 rpm. During this process, the added water was mixed for about 1 min. Next, sand was added and mixed for about 3 min. Finally, the Na_2SiO_3 solution was added to the mixture, followed by paddling for additional 2 min. The fresh geopolymer mortar was casted into the acrylic mould of $5 \times 5 \times 5$ cm in immediately after mixing in two layers. To compact the specimen, each layer was given 25 to 30 strokes by the rod bar, and then vibrated for 15 s on a vibrating table.

Furthermore, ordinary Portland cement type 1 was used to produce cement mortar in order to compare with geopolymer mortar, and the specimens were prepared in accordance with ASTM C 305 standard by using the weight ratio of water-to-cement (w/c) and the sand-to-cement ratio of 0.5 and 2.0, respectively.

2.4. Dielectric property measuring of constituents

In this study, the workability of the fresh geopolymer mortar was measured by applying the conventional flow and setting time tests of mortar cement. The details of these two test methods were explained in Sections 2.5 and 2.6, respectively.

2.5. Flow test

In this study, the flow table of geopolymer mortar was studied. Because the flow table test indicates the workability of the fresh mortar as according to ASTM C 109 standard. The workable flow of geopolymer mortar was in the range of $110 \pm 5\%$. The details of each mixture of geopolymer mortar for flow test are given in Table 1.

2.6. Setting time test

As for cement paste, the geopolymer mortar specimens with added water to meet flow values according to ASTM standard were tested for setting time by using the Vicat needle apparatus in accordance with ASTM C 191 standard.

In order to study the effect of the time after mixing before heat curing on dielectric properties geopolymer, the cast specimens were wrapped with vinyl sheet to prevent moisture loss and kept in laboratory at room temperature of 28–29 °C. Then the dielectric properties of geopolymer mortars were measured over 24 h after mixing. The detailed testing of the dielectric properties is explained in Sections 2.7.

2.7. Dielectric property measurement

In order to analyze the feasibility of using microwave energy application to cure geopolymer, the dielectric properties were studied to obtain the relative dielectric constant (ε_r') and the relative dielectric loss factor (ε_r''). These dielectric properties demonstrate the absorbability of microwave energy and the productivity of heating generated from microwave energy.

The open-ended probe technique was employed for measuring the dielectric properties of constituents and mortar of geopolymer. This technique, by itself, calculated the dielectric properties from the phase and amplitude of the reflected signal at the end of an open-ended coaxial line, which was inserted into a specimen measured by a portable network analyzer, as shown in Fig. 1. The analyzer consists of a coaxial cavity, microwave reflectometer, 0.35-cm coaxial cable, 0.35-cm female calibration, and short-and open-matched load and

Table 1
Mixture proportions of geopolymer mortar for flow test.

Specimen series	Ratios by weight		Weight (g)			
	Fly ash/activators (FA/AS)	Na_2SiO_3/NaOH (NS/NH)	Sand	Fly ash	Na_2SiO_3	NaOH
1) 2.0FA0.5	2.0	0.5	600	300	50	100
2) 2.0FA1.0	2.0	1.0	600	300	75	75
3) 2.0FA2.0	2.0	2.0	600	300	100	50
4) 2.5FA0.5	2.5	0.5	600	300	40	80
5) 2.5FA1.0	2.5	1.0	600	300	60	60
6) 2.5FA2.0	2.5	2.0	600	300	80	40

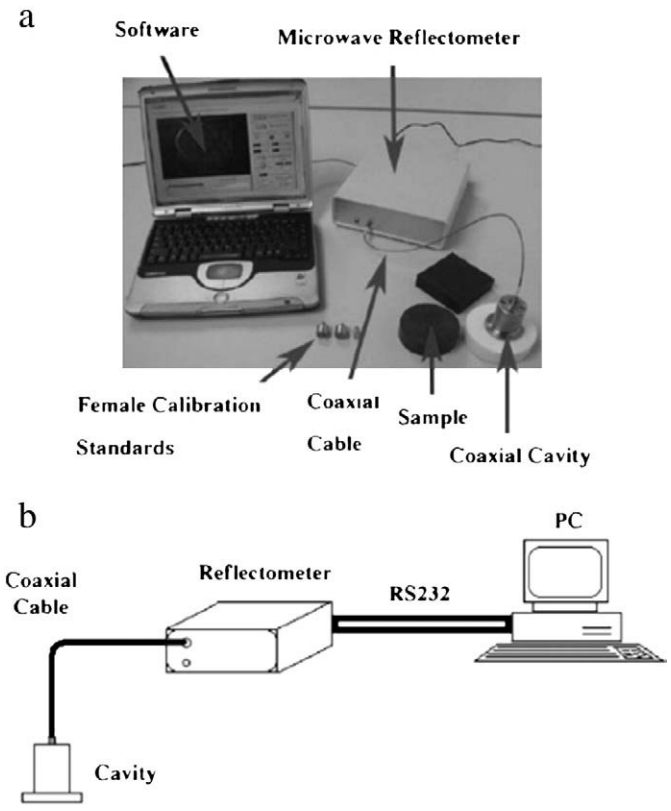

Fig. 1. Portable dielectric measurement (Network analyzer).

software. The coaxial cavity is characterized by a measurement range of 1.5–2.6 GHz with a precision of not more than 2% of the dielectric constant and 5% of the dielectric loss factor. The measured specimen should be assumed to be of infinite size and non-magnetic material, and have isotropic and homogeneous properties. In addition, the coaxial cavity must be in close contact to the specimen during the test. The five values of ε_r' and ε_r'' were averaged in order to represent the dielectric properties of each sample.

2.7.1. Dielectric properties measurement of geopolymer constituents

Since the dielectric constant (ε') of composite material is based on the weighted average of the constituents, this study examines not only the dielectric properties (ε_r' and ε_r'') of geopolymer mortars but also the dielectric properties of constituents. Each constituent was poured into the acrylic mold of $5\times5\times5$ cm. Then, dielectric properties (ε_r' and ε_r'') measurement were carried out at room temperature of 28–29°C.

2.7.2. Dielectric properties measurement of geopolymer mortar

Immediately after casting, the mortar specimens were measured for the dielectric properties (ε_r' and ε_r'') by using the coaxial probe technique and evaluating through the Network analyzer apparatus. Since, being able to cure at ambient temperature is very important in terms of practical application, the effect of time after mixing on the properties of geopolymer mortar were measured at time of 1, 2, 3, 4, 5, 6, 12 and 24 h.

2.8. Temperature measurement

After casting (time after mixing at 0 h), the surface temperature of geopolymer mortars are measured by using an infrared thermometer. This configuration facilitates temperature measurement from a distance without contact with the sample to be measured. Then, the test specimens and moulds were wrapped with vinyl sheet to prevent moisture loss and cured in ambient condition at room temperature (28–29 °C). After that the mortars were measured surface temperature again at time 1, 2, 3, 4, 5, 6, 12 and 24 h.

3. Results and discussion

In this section, the experimental results of geopolymer mortar which cover the effects of time after mixing and mixture proportion on the geopolymer mortar properties of fresh and hardened stage are presented and discussed. The flow characteristic and setting time were studied as the fresh properties of geopolymer and the results are explained in Sections 3.1 and 3.2, respectively. The results of dielectric properties of constituents and mortars of geopolymer at various times are shown in Sections 3.3 and 3.4, respectively. Finally, the variation in surface temperature of mortars with various mixture proportions and time after mixing are shown in Section 3.5.

3.1. Flow characteristic

The fresh fly ash-based geopolymer mortars have dark color and shiny appearance. Also, the mixtures are generally cohesive. Table 2 shows the flows of the mortar, it can be seen that all mortars were very stiff with no flow value and some specimens hardened immediately at right after the mixing. In order to obtain the workable geopolymer mortar in accordance with ASTM C 109 standard and have the right liquid volume to ensure constant workability, the added water was gradually adjusted into the mixture. This reason is in agreement with those reported by Chindaprasirt et al. [13] who claimed that adding water could improve workability of the mortar. The added water content of each mixture was calculated with the percentage of geopolymer paste (fly ash + NaOH + Na_2SiO_3) by weight. For the same of added water content, it is shown that the flow value of mixtures decrease with the increasing of the weight ratios of FA/AS and NS/NH. This result is in agreement with those reported by Sathonsaowaphak et al. [14] who found that the workability decreased with the increasing in the sodium silicate-to-sodium hydroxide ratio owing to the high viscosity of sodium silicate. Fig. 2 shows the effect of mixture proportion on the amount of added water to obtain the flow value in accordance with ASTM standard. It is shown that the amount of added water increases with the increment of the weight ratios of FA/AS and NS/NH. This is because the internal structure of geopolymer were developed rapidly and continuously with increasing amount of Si [15] and the geopolymer system has more reactive SiO_2 when Na_2SiO_3 is added, and this phenomenon results to the proper adjustment of Si/Al ratio to form geopolymerisation.

3.2. Setting time

As mentioned earlier in Section 3.1, the flow value of the specimens without added water were very stiff with no flow value. As observed by van Davidovits [16] who claimed that geopolymer cement hardens rapidly at room temperature. Fast setting is the

Table 2
Flow values of geopolymer mortars.

Specimen series	Flow (%)									
	Additional water (% of geopolymer paste)									
	0	3	5	8	10	13	15	18	20	23
1) 2.0FA0.5	42	112*	152	NA	–	–	–	–	–	–
2) 2.0FA1.0	27	61	110*	245	NA	–	–	–	–	–
3) 2.0FA2.0	NA	15	32	81	107*	139	NA	–	–	–
4) 2.5FA0.5	NA	23	67	113*	152	NA	–	–	–	–
5) 2.5FA1.0	NA	18	42	86	105*	151	NA	–	–	–
6) 2.5FA2.0	NA	NA	NA	19	34	75	105*	148	NA	–

* Flow (%) were in accordance with ASTM C 109.

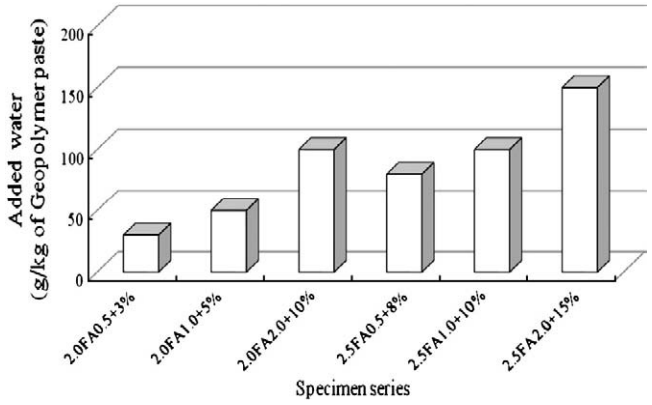

Fig. 2. Amount of required added water in geopolymer mortar for the flow to meet ASTM C 109 standard.

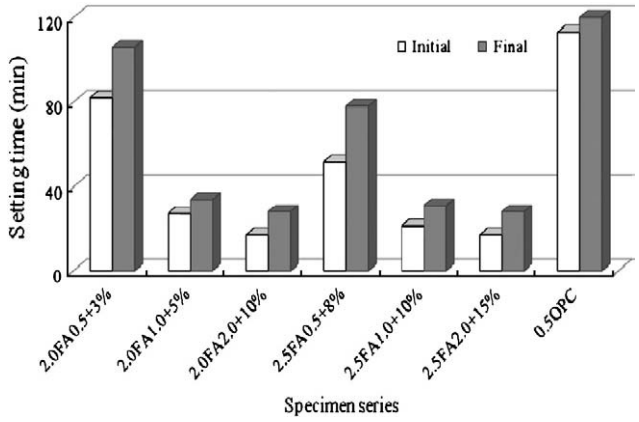

Fig. 3. Setting times of geopolymer mortars at various mixture proportions.

result of improved dissolution of the fly ash into alkaline liquid; leading to improved polymerisation and hardening of the gel phase [17]. Therefore this study only investigated setting time of the specimens with added water to reach the standard flow. The mixtures are given in Table 2 which have symbol of (*), namely 2.0FA0.5 + 3%, 2.0FA1.0 + 5%, 2.0FA2.0 + 10%, 2.5FA0.5 + 8%, 2.5FA1.0 + 10% and 2.5FA2.0 + 15%.

From the tested results, it shows that fly ash-based geopolymer mortars with added water were not hardened immediately at room temperature. As seen in Fig. 3, the initial and final setting times are hardly affected by the weight ratio of FA/AS and NS/NH. The setting time decreases when the weight ratio of FA/AS and NS/NH increase. For example, the 2.0FA0.5 + 3% and 2.5FA0.5 + 8% have initial setting times of 82 and 52 min, respectively, and the final setting times of 106 and 78 min, respectively. This result has been reported by Provis and van Deventer [18] that the specimens with higher weight ratio of solid materials-to-alkaline solution created more geopolymerisation than other specimens. The explanation of this result is that the weight ratio of FA/AS affects geopolymerisation reaction since this reaction requires SiO_2 and Al_2O_3 in fly ash [4]. The specimens with greater amount of fly ash have higher geopolymerisation reaction and rapid development of the internal structure. Thus, the setting time becomes lower.

3.3. Dielectric properties of constituents

Table 3 demonstrates the dielectric properties of constituents. The results show that constituents can be separated into two groups including the following:

(a) The water-based material group consists of water and alkaline solution (Na_2SiO_3 and NaOH (10 M)). It has a reasonably wide range of both properties (ε_r' and ε_r''). This is because water is a high lossy material corresponding to with ε_r' and ε_r'' much higher than that of the other components [19]. This effect shows clearly in alkaline solution that consists mainly of water and polymer.

(b) The solid material group comprises fly ash and sand (saturated surface-dry). This group shows a narrow range of dielectric constant (ε_r') and dielectric loss factor (ε_r''). The dielectric properties of this group are narrower than those of the water-based material group. This is caused by the facts that liquid constituents contain water is a polar molecule and the H–O–H bonding angle is not straight. This means that it can be easily polarized by an electric field and this is the main characteristic of a good dielectric [10]. But fly ash and sand have less polarity than that of water molecules, and thus become less affected by the forces generated by the alternating electromagnetic fields.

3.4. Dielectric properties of mortar

Consequently, the dielectric properties of geopolymer mortars are strongly affected by their constituents and mix proportion characteristics. In this section, the influences of time after mixing and mixture proportions on the dielectric properties of geopolymer mortars are discussed.

3.4.1. Effect of time after mixing

For dielectric properties of geopolymer mortar, the variations of ε_r' and ε_r'' with time after mixing are given in Figs. 4 and 5, respectively. It can be seen that both values of ε_r' and ε_r'' for all specimens are the highest at right after mixing. After that all the data show the decrease of the ε_r' and ε_r'' as a function of the time after mixing. This decreasing is strong at the 3 h first-geopolymerisation period and then slow down. Until the values of ε_r' and ε_r'' are the lowest at 24 h after mixing. This is because, in the early stages of the reaction, geopolymer composes of a large amount of free water and monomers as the main compositions [20] as shown in semi-schematic structure for geopolymer in Fig. 6. As mentioned in the early stages of the reaction, the speed of formation of dissolved monomers is greater than the speed of precipitation of the gel. The water and monomer are high lossy material corresponding to with ε' higher than that of the other components [10]. For any mixture, the values of ε_r' and ε_r'' decrease with increasing time, as expected. Because the free water and monomers in the early stages are transformed into bound water and polymer components, respectively which these components absorb microwave energy less than free water and monomer. And the bound water is tightly held and less rotationally free than the free water which makes higher dielectric losses possible [21]. In other words, the structure and chemical compositions of geopolymer are changed dramatically at the early stages of mixing process and decrease steadily after mixing time of 1 hour. However the ε_r'' of the mortar during a 1 hour first-period has a wide variation that is greater that its dielectric

Table 3
Dielectric properties of geopolymer mortar constituents.

Material	Relative dielectric constant (ε_r')	Relative dielectric loss factor (ε_r'')
Fly ash	2.90	0.21
Sand (Saturated surface-dry)	2.78	0.20
Sodium silicate (Na_2SiO_3)	8.94	1.70
Sodium hydroxide (NaOH, 10 M)	14.32	1.17
Water (27 °C)	72.18	11.75

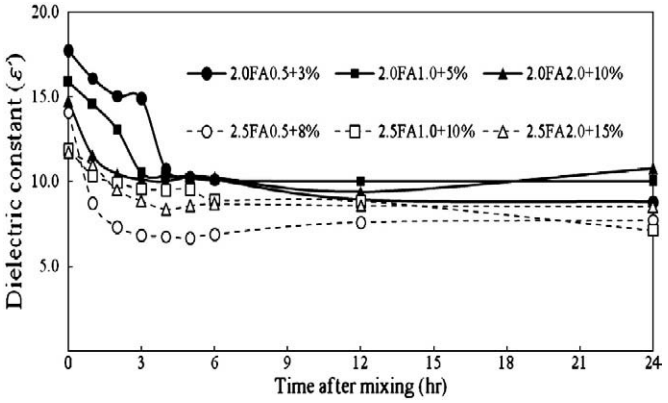

Fig. 4. Variation in dielectric constant with time after mixing.

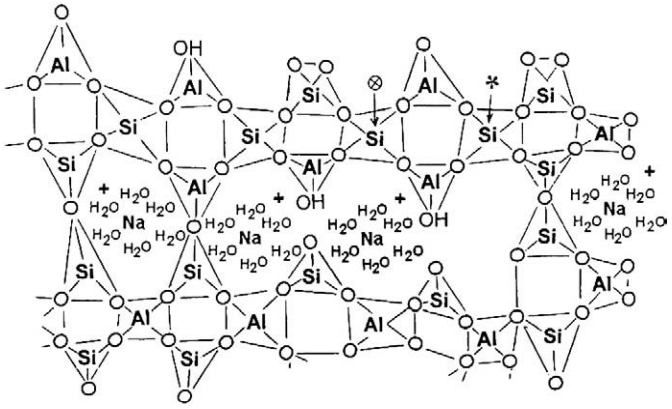

Fig. 6. Proposed semi-schematic structure for Na-polysialate polymer [14].

constant ε_r'; this is due to having a high variation and it is related to the conductivity of a material's polarization and relaxation behaviors [19]. By the physical (gas–liquid–solid) phase undoubtedly has an effect upon the dielectric properties of materials. Liquids may exhibit much higher values of dielectric constant and loss factor than if they are present in the solid state [22].

3.4.2. Effect of mixture proportions

Consideration of mixture proportions, it can be seen that at right after mixing, the increasing weight ratio of FA/AS and NS/NH decrease the ε_r' values of mortars. All specimens with the ratio of FA/AS of 2.0 have the ε_r' more than FA/AS of 2.5 specimens. These values have been shown to be correlated with liquid constituents content (added water and alkaline solution ($Na_2SiO_3 + NaOH$)). The amount of added water and alkaline solution that were used to produce geopolymer mortars as shown in Fig. 7. This is the ε_r' value of mortar with high liquid constituents content is higher than the mortar with less liquid content. This agrees with the results of constituents in Section 3.3 that shown liquid constituents contain water has the high values of dielectric properties. It is important to note that the dielectric properties of geopolymer mortars are strongly influenced by water content. The water content has a substantial effect on the final properties of the geopolymer [23]. At right after mixing, not only the ε_r' but also the ε_r'' values decrease with increasing the ratio of FA/AS while it not indicates clearly for variation of the ε_r'' values with the ratio of NS/NH. This is due to the variation of the chemical composition of reaction products and the complex of the internal structure of geopolymer. As the activation reaction rate as well as the chemical composition of the reaction products depend on several factors like for example the particle size distribution and the mineral composition of the starting fly ash, the type and concentration of the activator, etc. [24]. After 4 h of geopolymerisation period, it not shows clearly for variation of dielectric properties with the ratio of FA/AS and NS/NH. However, in next study, moisture measurement is important since the internal moisture transport is mainly attributable to capillary flow of liquid water through the voids during the initial stage of drying [25].

3.5. Temperature of mortars

Since geopolymerisation is exothermic [4], the release heat during its geopolymerisation is translated into an important temperature increase from which the heat quantity developed can be evaluated. The temperature evolution profile during a 24-hours geopolymerisation period at room temperature of all geopolymer mortars are displayed in Fig. 8. The results show that the temperature of mortars is the highest at right after mixing and decreased with time after mixing. This decreasing is strong at the 3 h first-geopolymerisation period and then slow down. This result is in agreement with those data from conduction calorimetry testing reported by Palomo et al. [26] that shows a rapid and intense release of heat when fly ashes and alkalis get in contact), as well as the analytical data published by van Deventer et al. (who found the existence of a type of synchronism correlating the Al and Si dissolution rate in a number of silicoaluminate minerals) [23], confirm for the alkali activation of fly ashes a similar process to that of hydration of Portland cement during the first moments of the hydration. For mixture proportions, it can be seen that at right after mixing, the increasing weight ratio of FA/AS and NS/NH decrease the temperature of mortars. These results agree with

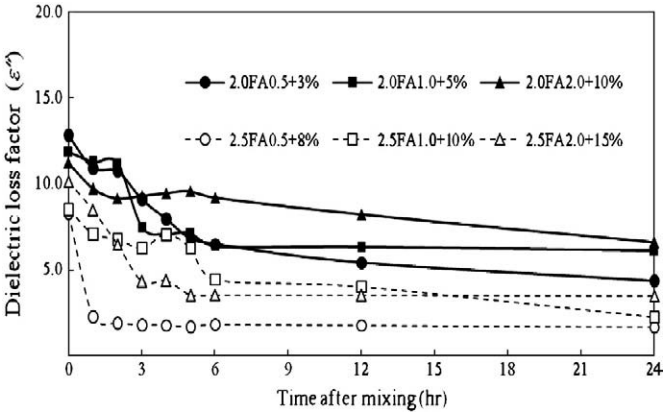

Fig. 5. Variation in dielectric loss factor with time after mixing.

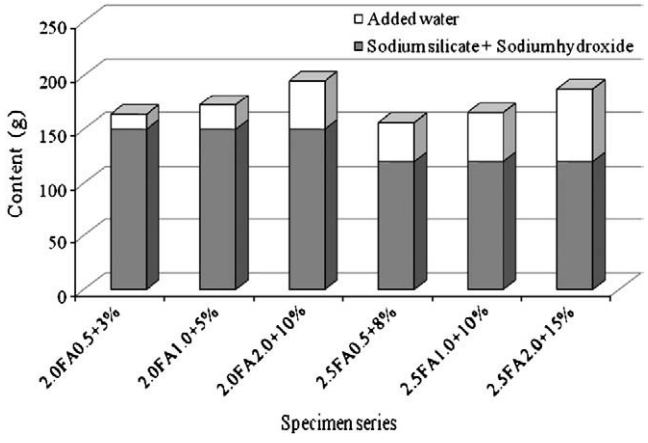

Fig. 7. Amount of added water and alkaline solution in geopolymer mortar.

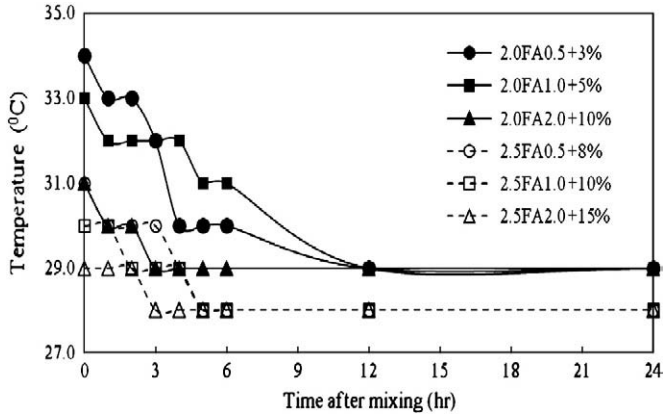

Fig. 8. Temperature evolution with time (0–24 h).

previous work that reported at high sodium silicate content, its effect became dominant and the effect of leaching of fly ash was less, thus it lead to decreased polymerization.

In addition, the results indicated that the variation of the surface temperature with time after mixing is similar to the variation of the dielectric properties (ε_r' and ε_r'') as shown in Figs. 9 and 10, respectively. However, in a previous report [27], the dielectric loss factor increased with increasing temperature. It is important to note that the variation of the dielectric constant with temperature is much less important than its variation with free water content [28–30].

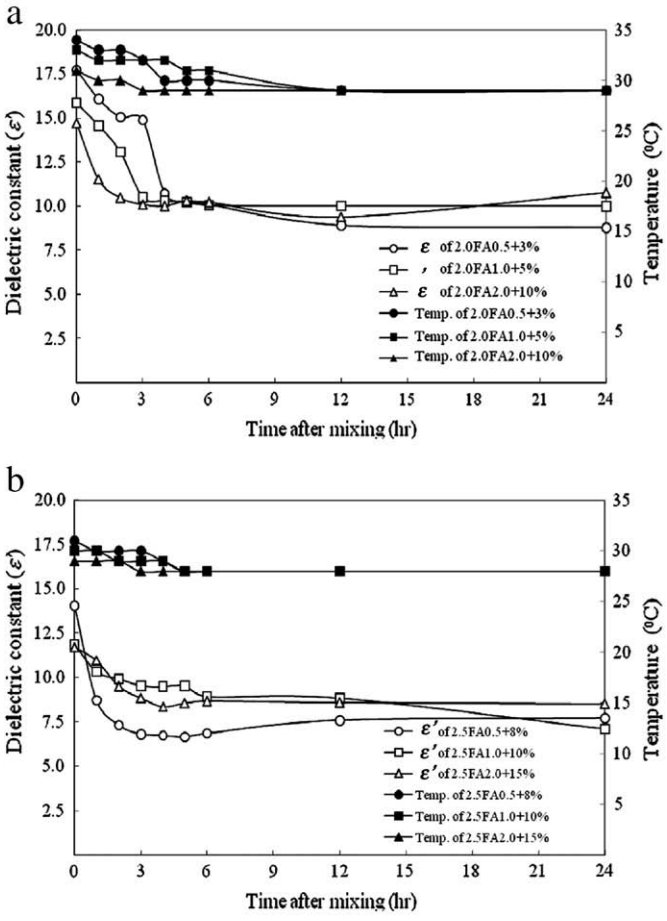

Fig. 9. Variation of dielectric constant and temperature with time after mixing. (a) Specimens with weight ratios of FA/AS equal to 2.0 and (b) Specimens with weight ratios of FA/AS equal to 2.5.

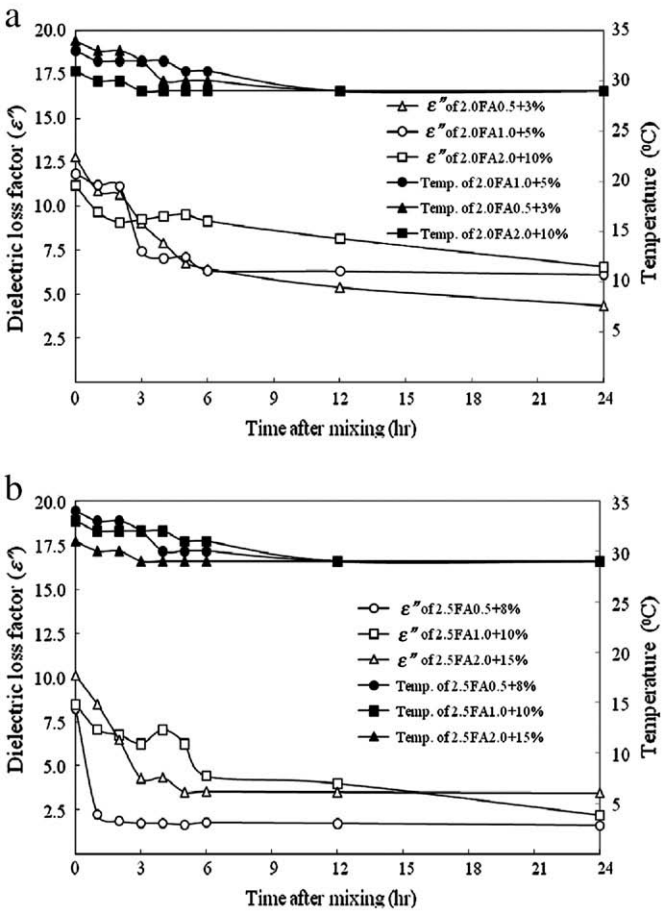

Fig. 10. Variation of dielectric loss factor and temperature with time after mixing. (a) Specimens with weight ratios of FA/AS equal to 2.0 and (b) Specimens with weight ratios of FA/AS equal to 2.5.

4. Conclusions

Based on the obtained results in this study, it can be concluded as follows,

4.1 Added water improves the workability of fresh geopolymer mortar and makes the flows of specimens pass the requirement of ASTM C 109 standard. The amount of added water to obtain the flow values in accordance with ASTM standard increases with the increasing of weight ratios of FA/AS and NS/NH.

4.2 Initial and final setting times of all geopolymer mortars are less than those of cement mortars. And the setting time of geopolymer mortar decreases with increasing weight ratio of FA/AS and NS/NH.

4.3 The dielectric properties (ε_r' and ε_r'') of geopolymer mortar constituents have been shown to be correlated with water content. This is the ε_r' and ε_r'' of liquid constituents are higher than solid constituents.

4.4 The ε_r' and ε_r'' of geopolymer mortars are affected by time after mixing and mixture proportions. The dielectric properties of mortars tend to decrease continuously with increasing time after mixing. For the mortars with more water and liquid constituent content, the dielectric constant and dielectric loss factor are also higher.

4.5 Measurement of dielectric properties of constituents and mortars of geopolymer show that the fly ash-based geopolymer mortars can be cured with microwave energy depending on the mixture proportion and time after mixing.

4.6 The temperature of mortars decrease with increasing time after mixing and weight ratio of FA/AS and NS/NH.

The results of this study are significant to a further research conducted along the similar line as well as to the applications related to the curing of geopolymer with microwave energy at various microwave power and duration.

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