



## Effect of Thai Kaolin on properties of agricultural ash blended geopolymers



Chayane Tipayasam<sup>a</sup>, Pimpawee Keawpapasson<sup>a</sup>, Parjaree Thavorniti<sup>b</sup>,  
Thammarat Panyathanmaporn<sup>b</sup>, Cristina Leonelli<sup>c</sup>, Duangrudee Chaysuwan<sup>a,\*</sup>

<sup>a</sup> Department of Materials Engineering, Faculty of Engineering, Kasetsart University, Thailand

<sup>b</sup> National Metal and Materials Technology Center (MTEC), Thailand

<sup>c</sup> Department of Materials and Environmental Engineering, Faculty of Engineering, University of Modena and Reggio Emilia, Italy

### HIGHLIGHTS

- We used kaolin to synthesize geopolymer cured at 80 °C for 24 h to be solidified.
- Metakaolin, kaolin mixed with bagasse ash and rice husk ash were used to synthesize geopolymers cured at ambient temperature.
- Geopolymers synthesized from kaolin mixed with either bagasse ash or rice husk ash can be solidified at ambient temperature.
- Geopolymers gave quite high compressive strength.

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### ABSTRACTS

Geopolymer has been developed as an alternative material to Portland cement. It is based on the polymerization of alkali activators and oxides of silicon and aluminium from pozzolans. Pozzolans are materials mainly  $\text{SiO}_2$  and/or  $\text{Al}_2\text{O}_3$  which reacts with  $\text{NaOH}$  and  $\text{Na}_2\text{SiO}_3$  to generate a three dimensional aluminosilicate structure. The clean and fine kaolin from south of Thailand transforms into metakaolin at 550–600 °C, as determined by DTA. In this research, kaolin was heated at 600 °C, 700 °C and 800 °C for 2 and 6 h to optimize its transformation into metakaolin as pozzolans for geopolymers preparation. Additionally, the as-received kaolin was exploited also as a pozzolan for geopolymers in order to reduce heat energy consumption. However, it was found that kaolin alone did not display sufficient pozzolanic behavior to form geopolymer at 7 days of age unless specimens were slightly warm cured. Therefore, it was blended with more active pozzolans such as bagasse ash and rice husk ash. Finally, the results of compressive strength and microstructures were investigated and discussed.

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### 1. Introduction

High energy consumption and  $\text{CO}_2$  emission are the general problems of ordinary Portland cement production. Nowadays, it is widely claimed that geopolymers are more sustainable building materials. If we consider a geopolymer as an aluminosilicate material with amorphous and/or semi-crystalline structures [1,2] we can easily observe how the chemical bonds are similar in both materials and hence their properties. In order to replace totally the ordinary Portland cement by geopolymers, we have to find a cheap and abundant raw material with lower energy intake than clinker. Kaolin which is the most interesting raw material for geopolymer preparation since its activation, soon after dehydration treatment at 550–600 °C to transform to metakaolin, is easily carried out in low cost alkali solution [1]. For the purpose to improve properties

of the final kaolin-based product, materials with pozzolanic behavior such as bagasse ash and rice husk ash [1,3,4] have been introduced. When pozzolans are combined with alkali solution, they can react at room or eventually slightly high temperatures and generate the ‘geopolymerization’ to obtain a material called ‘geopolymer’ like the hydration reaction of cement [1,5].

Previously, metakaolin was used to produce geopolymers that could be synthesized and solidified at room temperature with high compressive strength [1,4]. The results of thermal analysis stated that the endothermic dehydroxylation began at 550 °C and the crystalline kaolin transformed to amorphous metakaolin at temperature higher than 600 °C [1,4].

Kaolin is a kind of pozzolan that reacts with alkali solution but requires higher temperatures than metakaolin to be a hard geopolymer. Bagasse ash and rice husk ash both contain very high silica and are claimed as pozzolans that can form geopolymers which would be hardened at ambient temperature but they need high amount of alkali solution [1].

\* Corresponding author. Tel.: +66 2797 0999x2107; fax: +66 2942 8896.

E-mail address: [fengddc@ku.ac.th](mailto:fengddc@ku.ac.th) (D. Chaysuwan).

The objectives of this research were: (1) to elucidate the feasibility of cast geopolymer by using kaolin instead of metakaolin, in purpose of energy consuming reduction (2) to compare compressive strength and microstructure of resultant geopolymers from various pozzolans, e.g., kaolin, metakaolin bagasse ash and rice husk ash [6,7]. The metakaolin was acquired from kaolin calcination at various temperatures and times. The main alkali solutions used to activate pozzolans were sodium hydroxide (NaOH) and sodium silicate ( $\text{Na}_2\text{SiO}_3$ ).

## 2. Experimental

### 2.1. Raw materials preparation and characterization

Washed kaolin (K) from MRD-ECC Co., Ltd., in Ranong province, bagasse ash (BA) from Kaset Thai Co., Ltd., in Nakhon Sawan province, and rice husk ash (RHA) from a rice mill in Angthong province, were ground by a centrifugal ball mill until all of the fine particles passed through sieve 325 mesh#, i.e., <45  $\mu\text{m}$ . Sodium silicate solutions and 10 molar (10 M) sodium hydroxide solution were used as the alkali activators. The chemical composition and morphology of the raw materials were characterized by X-ray fluorescence (XRF) (Axiosmax-advanced, PANalytical, the Netherlands) and a scanning electron microscope (SEM) (JSM-6380, Japan). The thermal behavior of kaolin was evaluated by a differential thermal analysis (DTA) (DTA7, Perkin Elmer, England). The result of DTA analysis in Fig. 1 presented that dehydroxylation of kaolin occurred at approximately 560 °C to transform to metakaolin (MK). Therefore, kaolin was calcined at 600 °C, 700 °C and 800 °C for 2 and 6 h with a heating rate of 10 °C/min and then all of calcined kaolin were determined by X-ray diffraction (XRD) (X'pert, Philips, the Netherlands) in order to determine the optimum calcination condition. Cu K $\alpha$  radiation was used and the generator settings were a 40 kV excitation potential with a current of 35 mA for long fine focus tubes. The programs typically used were scans of 45 min long, extending from 5° to 80° 2 $\theta$  with step size 0.02°.

### 2.2. Geopolymer pastes and samples preparation

Kaolin and metakaolin geopolymer pastes were prepared in this research. The solution of  $\text{Na}_2\text{SiO}_3$  (Chemmin Corporation Ltd.,  $\text{Na}_2\text{O}:\text{SiO}_2 = 1:3.12$ ) and 10 M NaOH with the ratio of  $\text{Na}_2\text{SiO}_3:\text{NaOH}$  at 3:2 were premixed at least 1 day prior to use. The ratio of solid to alkali liquid was 60:40 by weight. From previous research, the quantity of alkali liquid affected the flow ability and the compressive strength of geopolymer, the strength increased with the alkali liquid increased and the percentage of alkali liquid was suitably used 40% [1]. The alkali solution was mixed with kaolin or metakaolin as pozzolan solids and stirred to obtain a homogeneous paste. Consequently, the paste was poured into  $25 \times 25 \times 25 \text{ mm}^3$  acrylic moulds to produce specimens. Geopolymers of metakaolin specimens were cured in moulds at room temperature for 24 h and they were successfully produced. On the other hand, kaolin geopolymers were not perfectly solidified unless they were cured at 80 °C for 24 h. Then all specimens were removed from moulds and wrapped again with plastic film to be cured further at room temperature until the day of tests. Due to slow setting of kaolin geopolymers, then bagasse ash and rice husk ash were chosen to blend with kaolin in order to study their effects. The Si/Al and Na/Al molar ratio of the mixtures are listed in Table 2. The compressive strength tests were investigated at the age of 7, 14, 21, 28 and 91 days. The test was done according to the ASTM C 618. The reported results are the average of four samples. The microstructure of fracture surface of samples at the age of 28 days was

determined by SEM with an accelerating voltage of 15 kV, a probe current of 15 mA and a working distance of 10 mm. Gold coating was used for high resolution imaging in the SE-mode to display fine structure in the samples.

## 3. Results and discussions

### 3.1. Characterization of raw materials

The chemical compositions of raw materials are shown in Table 1, especially bagasse ash and rice husk ash consisting of very high  $\text{SiO}_2$ . DTA curve of kaolin is shown in Fig. 1, a broad endothermic peak at about 560 °C and a sharp exothermic peak at 980 °C were found. The endothermic peak was due to the dehydroxylation at 560–650 °C indicated that kaolin ( $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$ ) was transformed to metakaolin ( $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ ).

From XRD patterns of kaolin and various metakaolins, in Fig. 2, they showed sharper peaks of kaolin comparing to those of metakaolins. It was observed that kaolin mainly composed of kaolinite. Metakaolin patterns possessed peaks of the muscovite obviously at 8° and 26° 2 $\theta$ . Metakaolin, transformed from kaolin in all treatments, appeared more amorphous patterns with lower intensity and some peak disappeared, e.g., kaolinite because the dehydroxylation took place.

In Table 1, all pozzolans were characterized by XRF to clarify the content of  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$  and calculate further for Si/Al and Na/Al ratios (as in Table 2). The Si/Al ratios affect the compressive strength and microstructure of geopolymer [8]. When  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$  in pozzolans reacted with alkali solution, the geopolymerization networks of poly-sialate ( $\text{M}_n(-(\text{SiO}_2)_z-\text{AlO}_2)_n, \text{wH}_2\text{O}$ ) [9] would be formed depending on the Si/Al and Na/Al molar ratios. The Si/Al molar ratio of between 1 and 2 and Na/Al molar ratio of less than 1 were appropriate to produce geopolymers [1].

Table 2 showed the formulae of geopolymer with varied content of pozzolans as well as the Si/Al and Na/Al molar ratios. The meaning of each formula was, for example, the 100 K = 100% kaolin mixed with alkali solution and 90K10BA = 90% kaolin and 10% bagasse ash mixed with alkali solution.

### 3.2. Microstructure of raw materials

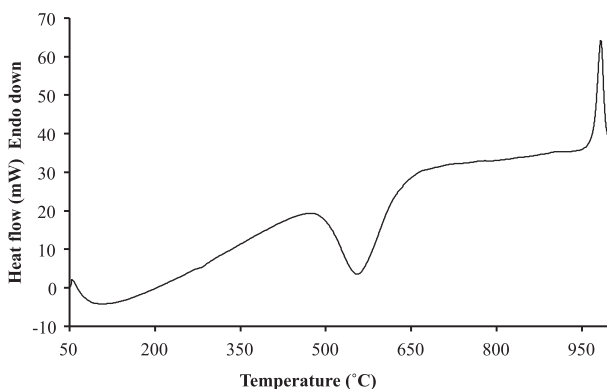
The SEM micrograph of kaolin (Fig. 3(a)) revealed that it formed of aggregates of small grains of sizes <45  $\mu\text{m}$ , with the laminar structure characteristic of these materials. Fig. 3(b) showed that the morphology of MK possessed coarser aggregates with irregular particles with respect to that of K as consequence of calcination process. The SEM morphology of processed BA showed the irregular shape of the particles that were also partly agglomerated (Fig. 3(c)) as consequence of the burning process. The microstructure of ground RHA, shown in Fig. 3(d), was found to present angular and fluff-like particles with the median size of 45  $\mu\text{m}$ .

### 3.3. Compressive strength test

In preliminary compressive strength results, on the different type of MK geopolymers at 7 days age (Fig. 4) it could be noted that all specimens presented values well above 29 MPa.

**Table 1**  
The chemical compositions of K, MK, BA and RHA determined by XRF.

	$\text{SiO}_2$	$\text{Al}_2\text{O}_3$	$\text{Fe}_2\text{O}_3$	CaO	MgO	$\text{Na}_2\text{O}$	K $_2\text{O}$	$\text{SO}_3$
K	51.21	43.82	1.70	0.04	0.19	–	1.95	0.01
MK	53.39	43.98	0.63	0.04	0.08	–	0.86	0.02
BA	75.39	5.44	3.42	7.89	1.50	0.30	3.32	0.39
RHA	94.25	0.52	0.22	0.70	0.40	0.05	2.26	0.82



**Fig. 1.** DTA pattern of washed kaolin.

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