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Research Paper

# Compressive strengths of water treatment sludge-fly ash geopolymer at various compression energies

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**ABSTRACT**

Sludge-fly ash geopolymer is a green material, which does not require cement as a cementitious binder. Sludge and fly ash (FA) are both by-products from Bang Khen water treatment plants and Mae Moh power plants, respectively. A liquid alkaline activator, L is a mixture of sodium silicate solution ($Na_2SiO_3$) and sodium hydroxide solution (NaOH). This article investigates the influence of compression energy (*E*) on the Unconfined Compressive Strength (UCS) of a novel sludge-fly ash geopolymer. The optimal liquid alkaline activator (L) content, sodium silicate solution to sodium hydroxide solution ratio ($Na_2SiO_3$/NaOH), heat temperature (*T*) and heat duration (*D*), providing the maximum UCS for each *E* is also investigated. Test results indicate that the optimal L/FA ratio decreases as *E* increases. The optimal L/FA ratios for *E* = 592.5, 1346.6 and 2693.3 kJ/m$^3$ are 1.5, 1.4 and 1.3, respectively. The $Na_2SiO_3$/NaOH ratio of 80:20 is considered as optimal for all *E* tested. The UCS of sludge-FA geopolymer at all *E* values tested increases with an increase of heat duration until a heat duration of 72 hours, after which the UCS becomes almost constant. The heat temperature accelerates the geopolymerization reaction and therefore enhances the strength. However, overheating results in micro-cracks in the samples due to the loss of moisture. As such, the optimal temperature is controlled by liquid content; the higher L/FA results in the lower optimal heat temperature. Since the optimal L/FA ratio decreases as *E* increases, the optimal *T* subsequently decreases with an increase in *E*; i.e., its values are 75, 85 and 95 $^oC$ for *E* = 2693.3, 1346.6 and 592.5 kJ/m$^3$, respectively. The maximum UCS values of sludge-FA geopolymer at optimal ingredient and heat condition are 20, 18 and 16 MPa for *E* = 2693.3, 1346.6 and 592.5 kJ/m$^3$, respectively, which meet the strength requirement of bearing masonry units as specified by the Thailand Industrial Standard (TIS).

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## 1. Introduction

Water production requires the extraction of water from natural sources. The water treatment process results in an ultra-soft sludge by-product. The clarifier system employed in water treatment plants results in the sludge flocculating and falling in the bottom of the treatment tank. The liquid sludge is subsequently drained to sludge lagoons for disposal. The increasing demand of treated water produced by the Metropolitan Waterworks Authority of Thailand (MWA) and in similar water treatment plants worldwide, has resulted in increasing quantities of sludge by-products generated annually. For MWA, the water treatment sludge is generated with the maximum capacity of 300 ton per day in the dry season and about 700 ton per day in the wet season. With a rapid increase in demand due to growing population, the quantity of water treatment sludge in MWA is increasing at ever increasing rate and hence the urgent need to find a sustainable reuse option for the growing stockpiles of sludge, which in the past have been disposed to landfills (Suksiripattanapong et al., 2014).

Alkali-activated alumino-silicate cement, known as 'geopolymers' has become increasingly popular in recent years as an environmental-friendly alternative to ordinary Portland cement (Davidovits 1991). Geopolymers are furthermore touted for their high performance (high strength and durability), low $CO_2$ emission and low energy consumption. Silica rich materials such as clay or kaolin (Kaps and Buchwald 2002), fly ash, and bottom ash (Davidovits et al. 1999) can be used as a precursor to react with the liquid alkaline activator.

Fly ash (FA) derived from coal-fired electricity generation provides the greatest opportunity for commercial utilization of this technology due to the plentiful worldwide raw material supply (Mohapatra and Rao 2001; Van Jaarsveld et al. 1998). Palomo et al. (1999) found that the different FA activated with 8-12 M NaOH cured at 85$^{o}$C for 24 hours produced a material with compressive strength of 35-40 MPa and about 90 MPa if sodium silicate ($Na_2SiO_3$) is added to the NaOH solution. Xie and Yunping (2001) reported that the hardening process of FA activated with $Na_2SiO_3$ is mainly attributed to the gel-like reaction products that bind FA particles together. FA is extensively used as a precursor for geopolymers in Australia (Rickard et al. 2011; Rickard et al. 2012) and Thailand (Chindaprasirt et al. 2001; Phetchuay et al. 2014; Sata et al. 2012).

Sukmak et al. (2013a; 2013b) previously investigated the possibility of using FA as a precursor and silty clay as aggregates to develop the clay-FA geopolymer masonry units. The 7-day strength of the clay-FA geopolymer is greater than 10 MPa, suitable as bearing masonry unit according to the Thailand Industrial Standard (TIS). The strength requirement is 2.5 MPa for non-bearing and 7.0 MPa for bearing masonry units (Horpibulsuk et al., 2014). The durability against sulfate attack of clay-FA geoplymer is better than that of clay-cement; there is no major change in the microstructure and pH of clay-FA geopolymer when exposed to sulfate solutions (Sukmak et al. 2015).

Recently, Suksiripattanapong et al. (2015) have investigated the strength development in water treatment sludge-FA geopolymer. The optimal ingredient providing maximum unit weight and strength is $Na_2SiO_3$ ratio of 80:20 and L/FA ratio of 1.3, irrespective of heat condition and curing time. The optimal heat temperature and duration for the optimal ingredient are 75$^{o}$C and 72 hours, respectively. The durability against wet-dry of sludge-FA geoplymer is found to be better than that of sludge-cement (Horpibulsuk et al. 2015). These studies are limited to a specified compression energy, while the masonry units can be manufactured at various compression energies.

This research aims to mainly investigate effects of compression energy, *E*, on the Unconfined Compressive Strength (UCS) development of water treatment sludge-FA geopolymer to ascertain its performance as a bearing masonry unit while the development of mix design method is not a focus of this paper as it is more of interest for masonry suppliers. The optimal ingredient (L/FA and $Na_2SiO_3$/NaOH ratio) and heat conditions (temperature, *T* and duration, *D*), which provide the maximum unit weight and UCS of the sludge-FA geopolymer for each E is also examined and presented. The microstructural observation of sludge-FA geopolymer via scanning electron microscope (SEM) is furthermore undertaken to understand the role of *E* on the UCS development. This research will enable sludge traditionally destined for landfill to be used in a sustainable manner as an aggregate in geopolymer masonry units, which is significant from engineering, economical and environmental perspectives.

## 2. Materials and methods

### 2.1 *Materials*

The sludge was obtained from the Bang Khen water treatment plant of Metropolitan Waterworks Authority, Bangkok, Thailand. The sludge consists of 0.15, 99.65 and 0.20 percent of sand, silt and clay, respectively. The grain size distribution, mineral and chemical compositions of the sludge were obtained from laser particle and X-ray Fluorescence (XRF) analysis as shown in Fig. 1 and Table 1, respectively. The sludge is

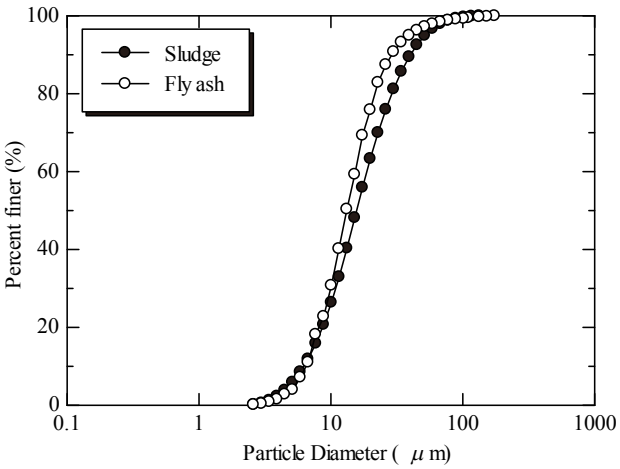


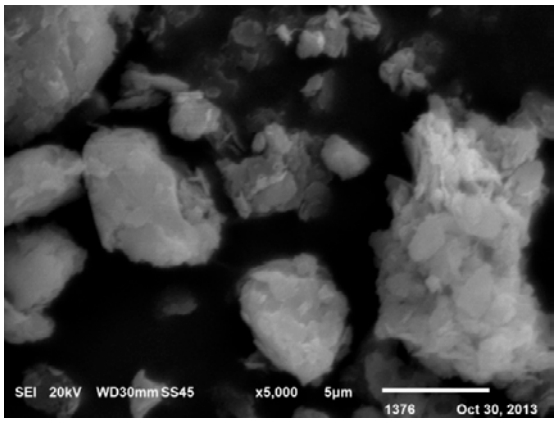

**Fig. 1.** Grain size distribution of sludge and FA.

classified as non-plasticity with the specific gravity of 2.60.

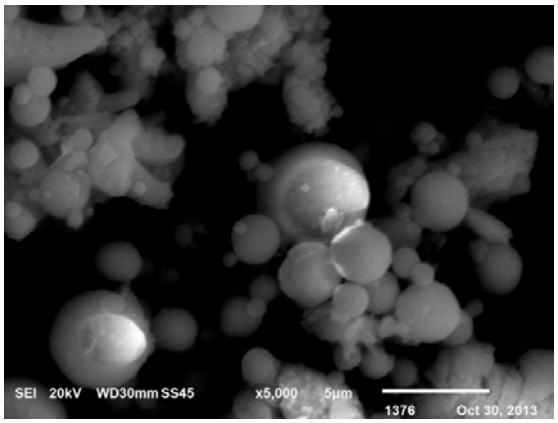
FA was obtained from the Mae Moh power plant in the northern region of Thailand. Table 1 summarizes the chemical composition of FA using XRF. Total amount of the major components ($SiO_2$, $Al_2O_3$ and $Fe_2O_3$) are 67.31% and CaO content is 30.24%; therefore, it is classified as a Class C fly ash. Figure 1 shows the grain size distribution curve of FA, as determined by Laser particle size analysis. It is shown that the sludge particles are similar to FA ones. The average grain size of FA is 13.25 $\mu$m. The specific gravity of FA is 2.35. The morphology of the sludge and the FA is shown in Fig. 2. The FA particles are fine and spherical whereas the sludge particles are irregular in shape. The liquid alkaline activator (L) is a mixture of $Na_2SiO_3$, which consists of 9% $Na_2O$ and 30% $SiO_2$ by weight, and NaOH with a concentration of 10 molars.

### 2.2 *Sample preparation*

The sludge-FA geopolymer sample is a combination of sludge, FA and liquid alkaline activator ($Na_2SiO_3$ and NaOH) by weight. The sludge/FA ratio was fixed at 70:30 and the $Na_2SiO_3$/NaOH ratios were 100:0, 90:10, 80:20, 70:30 and 50:50 as previously suggested by Suksiripattanapong et al. (2015). The compression energies, *E,* were set as 592.5, 1346.6 and 2693.3 kJ/m$^3$, corresponding to standard Proctor, half-modified Proctor and modified Proctor energies, respectively. The test results at the modified Proctor energy obtained from Suksiripattanapong et al. (2015) were taken and analyzed in this study. The air-dried sludge and FA were mixed for 5 minutes in a mixer to ensure homogeneity of the mixture. The mixer was stopped and the mixture was activated by the liquid alkaline activator and mixed for additional 5 minutes. The mixture was then statically compressed in a cylindrical mold with 50 mm in diameter and 100 mm in height. Compression was performed at the optimum water content with a manual hydraulic jack to attain the maximum unit weight. Both optimum water content and maximum unit weight were obtained from Proctor compaction tests at half standard, standard, half modified and modified Proctor energies. The samples were dismantled, wrapped within vinyl sheet and then cured at 65, 75, 85 and 95$^o$C for 24, 48, 72, 96 and 120 hours. After the temperature curing, the samples were cured at room temperature (27-30$^o$C) until lapse of different curing times as planned.




(a) Sludge




(b) Fly ash (FA)

**Fig. 2.** SEM of sludge and FA.

**Table 1.** Chemical composition of sludge and fly ash.

| Chemical composition (%) | Sludge | Fly ash |
|---|---|---|
| $SiO_2$ | 61.84 | 47.51 |
| $Al_2O_3$ | 24.80 | 13.14 |
| $Fe_2O_3$ | 9.52 | 6.66 |
| CaO | 0.60 | 30.24 |
| MgO | N.D. | N.D. |
| $SO_3$ | 0.59 | N.D. |
| $Na_2O$ | N.D. | 0.41 |
| K2O | 1.90 | 1.63 |
| LOI | 0.75 | 0.42 |

Note: N.D. = Not detected

UCSs of sludge-FA geopolymer samples were measured after 7 days of curing. The growth of the geopolymerization structures on the samples was illustrated using Scanning Electron Microscope (SEM). The sludge-FA geopolymer samples were carefully broken and small fresh fragments were taken from the center for SEM tests. The SEM samples were frozen at -195°C by immersion in liquid nitrogen for 5 minutes and evacuated at a pressure of 0.5 Pa at -40°C for 5 days (Du et al. 2014a, b; Sukmak et al. 2013a; Sukmak et al. 2013b; Horpibulsuk et al. 2010). All samples were coated with gold before the SEM (JOEL JSM-6400) analysis.

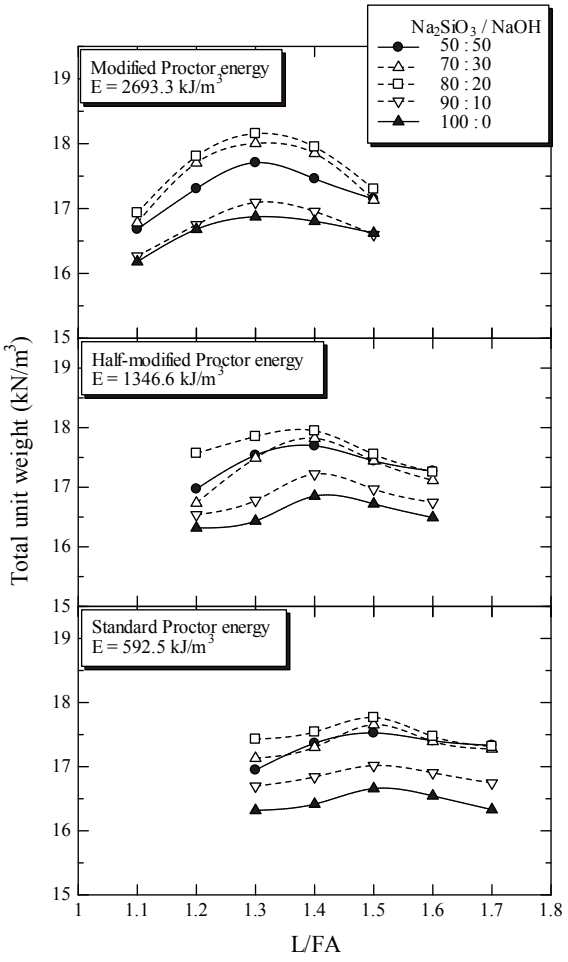



**Fig. 3.** Compaction curves of the sludge–FA–L mixture for different compaction energy.

## 3. Test results and discussion

Figure 3 shows the relationships between unit weight versus L/FA ratio at various $Na_2SiO_3$/NaOH ratios and *E* of sludge-FA geopolymer. Test results indicate that the unit weight of sludge-FA geopolymer increases with increasing L/FA ratio until the maximum unit weight is attained at an optimal L/FA value. Beyond this optimal value, the unit weight decreases as the L/FA ratio increases. The optimal L/FA ratio decreases as *E* increases. The optimal L/FA ratios for *E* = 592.5, 1346.6 and 2693.3 kJ/m$^3$ are 1.5, 1.4 and 1.3, respectively. This increase in L/FA ratio is in agreement with the study reported by Horpibulsuk et al. (2008; 2009) for compacted soils, whereby the optimal water content decreases with increasing compression energy. The decrease in maximum unit weight is associated with the increase in L/FA ratio. For all *E* values tested, the maximum unit weight increases with increasing $Na_2SiO_3$/NaOH until the $Na_2SiO_3$/NaOH ratio is equal to 4.0 ($Na_2SiO_3$/NaOH = 80:20). Beyond this value, the maximum unit weight decreases. In other words, the $Na_2SiO_3$/NaOH ratio of 4.0 gives the highest maximum unit weight, regardless of *E*.

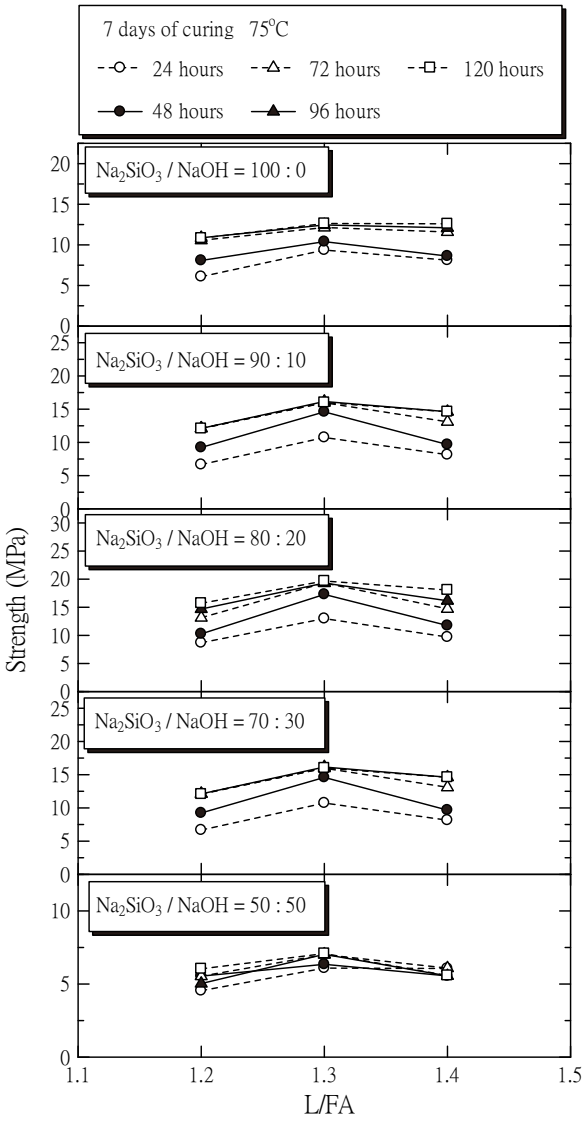



**Fig. 4.** 7-day strengths of the sludge–FA geopolymer samples heated at 75°C for L/FA ratios of 1.2, 1.3 and 1.4 and modified Proctor energy.

The effect of L/FA ratio on UCS development in sludge-FA geopolymer is illustrated in Figure 5, which shows the relationships between 7-day UCS versus L/FA ratio for the $Na_2SiO_3$/NaOH ratios of 100:0, 90:10, 80:20, 70:30 and 50:50. The samples were heated at 75 °C for 24, 48, 72, 96 and 120 hours and compressed at modified Proctor energy (2693.3 kJ/m$^3$), respectively. The test result shows that the maximum UCS values of sludge-FA geopolymers are at L/FA ratio of 1.3 for all the $Na_2SiO_3$/NaOH ratios. The UCS results on the wet and dry sides of the optimum L/FA are similar. The $Na_2SiO_3$/NaOH ratio of 80:20 gives the maximum UCS for all L/FA ratios. It is evident from Figs. 3 and 4 that the optimal $Na_2SiO_3$/NaOH ratio, providing the maximum UCS and unit weight, is essentially the same for all *E* values and is 80:20, while the optimal L/FA ratio varies and is dependent upon *E*.

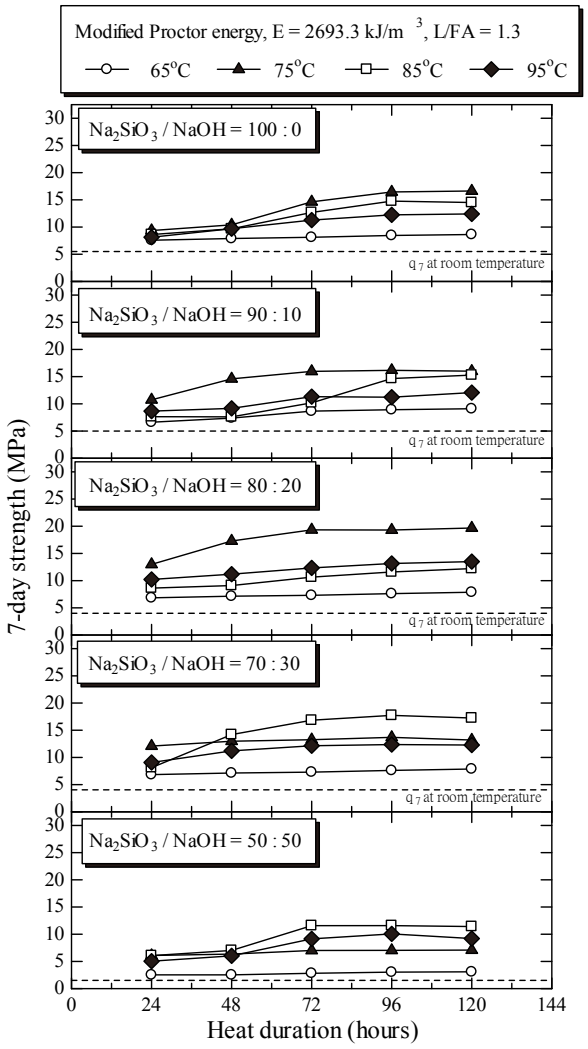



**Fig. 5.** Effects of heat condition on the 7-day UCS for modified Proctor energy.

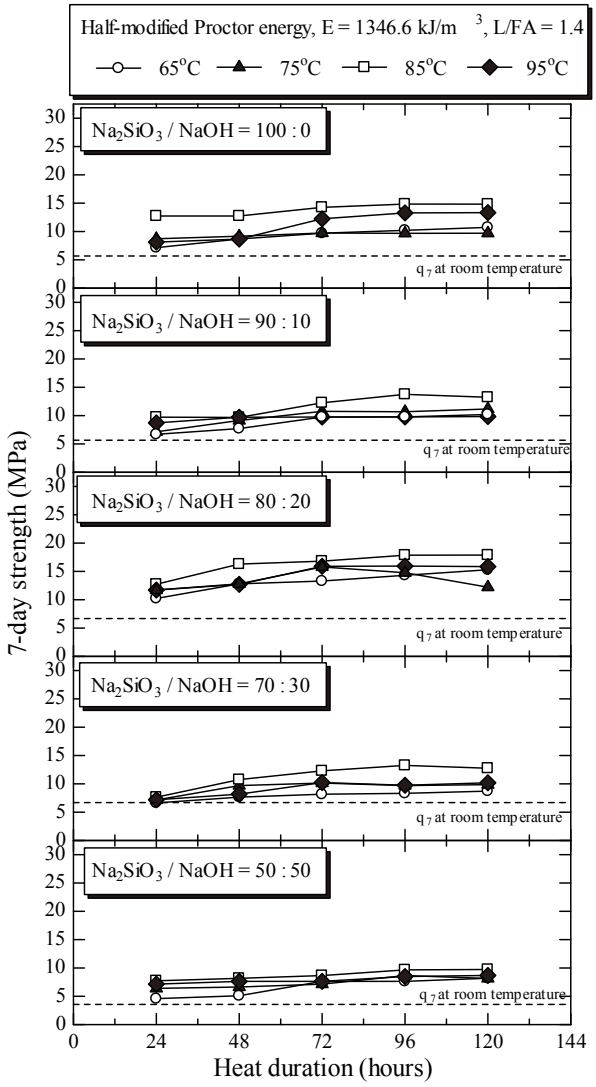



**Fig. 6.** Effects of heat condition on the 7-day UCS for half-modified Proctor energy.

Figures 5-7 indicate the relationship between 7-day UCS versus heat duration, *D* of sludge-FA geopolymer samples for various heat temperatures (room temperature, 65, 75, 85 and 95°C), $Na_2SiO_3$/NaOH ratios (50:50, 70:30, 80:20, 90:10 and 100:0), and compression energies (592.5, 1346.6 and 2693.3 kJ/m$^3$). For all of the compression energies and L/FA ratios, the UCS of sludge-FA geopolymer samples increases as *D* increases until a threshold heat duration of 72 hours, after which the UCS retains almost constant. The optimal *T*, providing the highest strength is found to be dependent upon *E* and is 75°C, 85°C and 95°C for *E* = 2693.3, 1346.6 and 592.5 kJ/m$^3$, respectively. This UCS development is noted to be different from the previous study of Sukmak et al. (2013a; 2013b) for clay-FA geopolymer, whereby the UCS increases to a peak value and subsequently decreases as *D* increases. In other

words, the sludge-FA geopolymer exhibits a more stable structure (insignificantly sensitive to change in water content due to heat) than the clay-FA geoploymer, which can be attributed to the sludge being non-plastic. The long-term strengths were not measured in this study. However, based on the authors previous research on sludge-fly ash geopolymer (Suksiripattanapong et al. 2015), the compressive strength of sludge-FA geopolymer tends to be constant after 7 days of curing. It is because the heat curing accelerates the geopolymerization reaction and hence the high early strength with insignificant strength development over curing time.

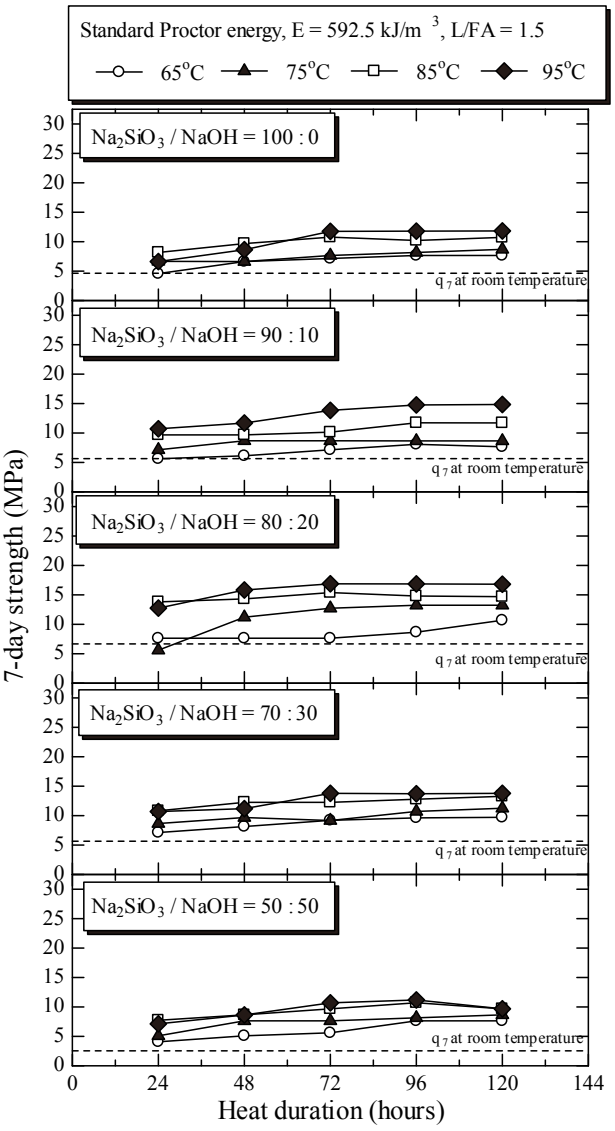



**Fig. 7.** Effects of heat condition on the 7-day UCS for standard Proctor energy.

Figure 8 shows SEM images of 7-day cured geopolymer samples at optimum ingredient ($Na_2SiO_3$/NaOH ratio of 80:20 and L/FA ratios of 1.5, 1.4 and 1.3 for $E$ = 592.5, 1346.6 and 2693.3 kJ/m$^3$, respectively) at various $T$ (room temperature, 75 $^o$C and 85 $^o$C) for 72 hours (optimal duration). The chemical attack on the FA surface at room temperature is insignificant for all $E$ values while the etching on the FA surface and geopolymerization products filling in the pore space is clearly seen at higher temperatures of 75 and 85$^o$C.

The optimal heat temperature for each $E$ is examined using SEM images (Fig. 8). For $E$ = 2693.3 kJ/m$^3$, the overheating (85 $^o$C) causes the micro-cracks due to the loss of the pore fluid, which agrees with previous studies by Sukmak et al. (2013a; 2013b). As such, the highest 7-day UCS of the sludge-FA geopolymer is obtained at 75$^o$C, where the SEM image clearly shows the geopolymerization products in the pore space and on FA surface. For $E$ = 1346.6 kJ/m$^3$, the geopolymerization products are clearly observed on FA particles and pore space at $T$ = 85 $^o$C; this temperature is considered as optimal and provides the highest compressive strength (Fig. 6). Since the micro-cracks on the microstructure of the geopolymer is due to the loss of moisture, the optimal temperature is controlled by liquid content in the geopolymer samples; i.e., the higher L/FA ratio results in the lower optimal heat temperature. Consequently, the optimal $T$ decreases with an increase in $E$; i.e., its values are 75, 85 and 95 $^o$C for $E$ = 2693.3, 1346.6 and 592.5 kJ/m$^3$, respectively.

Figure 9 shows the SEM images of 7-day cured geopolymer samples constituted at $Na_2SiO_3$/NaOH ratio = 80:20, and at optimum L/FA ratios and heat temperatures (optimal L/FA ratios = 1.3, 1.4 and 1.5 and optimal $T$ = 75, 85 and 95$^o$C for $E$ = 2693.3, 1346.6 and 592.5 kJ/m$^3$, respectively) for various heat durations. The geopolymerization process and the growth of geopolymerization products at various heat durations (24, 72 and 120 hours) are clearly observed. The alkaline dissolution results in etching on the FA surface within 24 hours of heating for all samples. The etched holes on the FA surface are then filled with smaller FA particles and welded by the geopolymerization products after 72 hours (position A). The observation of the growth of geopolymerization products with heat duration is similar to that reported by Palomo et al. (1999) for FA-geopolymers (without aggregate). Even though the growth of geopolymerization products still continues even after 72 hours, the micro-cracks developed due to loss of moisture retard the UCS development. Consequently, the UCS of the sludge-FA geopolymer increases insignificantly after 72 hours (Figs. 5, 6 and 7).

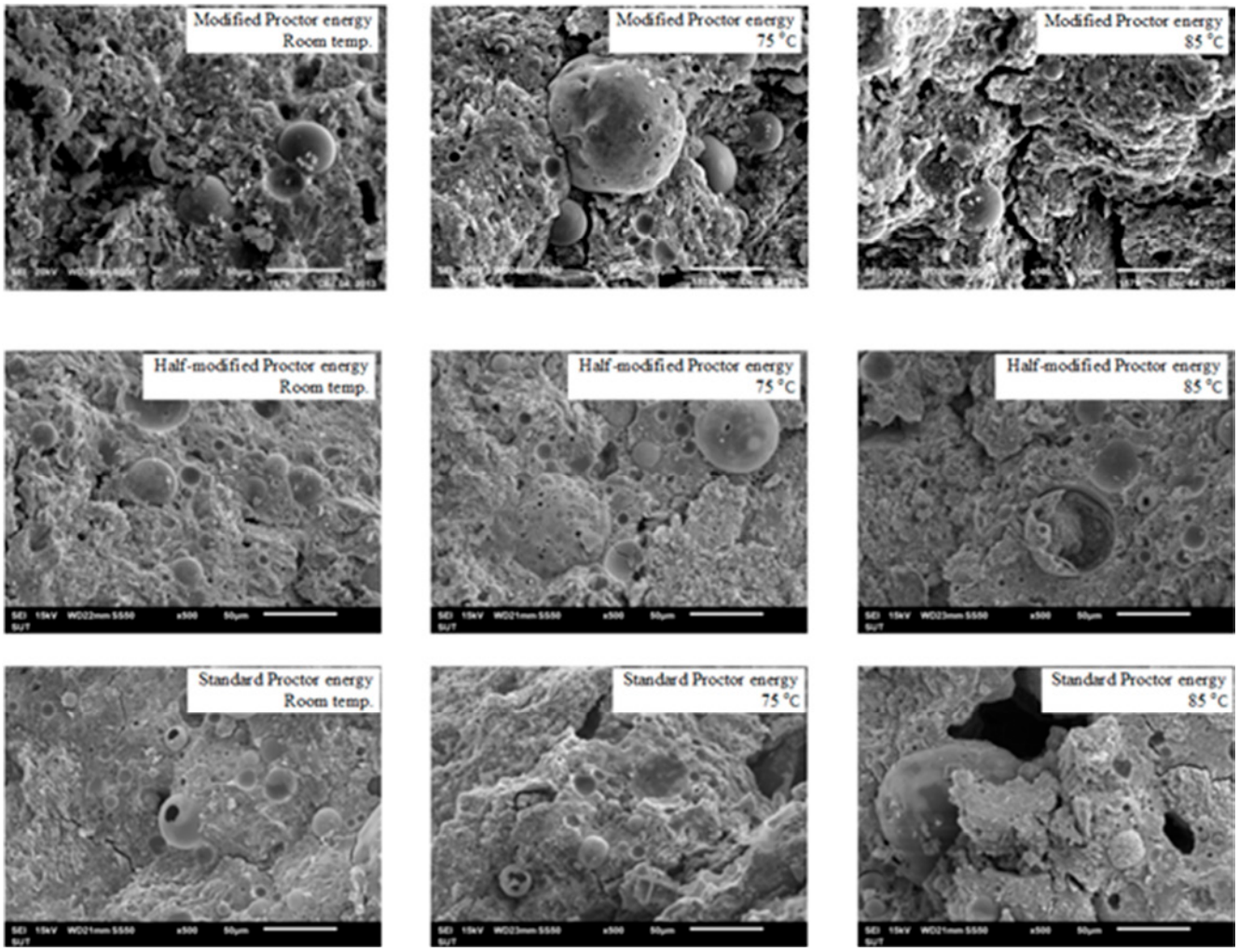



**Fig. 8.** SEM images of the 7-day cured sludge-FA geopolymer at $Na_2SiO_3$/NaOH ratio of 80:20 and at various compression energies and temperatures for 72 hours.

## 4. Conclusions

The effect of compression energy, *E* on the UCS development of the sludge-FA geopolymer is researched in this study. The optimal ingredients (L/FA ratio and $Na_2SiO_3$/NaOH ratio), and heat condition (heat temperature and duration), which provides the maximum unit weight and UCS under various compression energies are also investigated. The Scanning Electron Microscopy (SEM) analysis is undertaken to understand the effect of *E* on the UCS development. The key findings of the research are summarized as follows.

1. For a particular *E*, the unit weight and UCS of sludge-FA geopolymer increase with increasing L/FA ratio until the maximum unit weight and UCS are attained at an optimal L/FA ratio. Beyond this optimal value, the unit weight and UCS decrease as the L/FA ratio increases. The optimal L/FA providing the maximum unit weight and UCS are approximately 1.5, 1.4 and 1.3 for *E* = 592.5, 1346.6 and 2693.3 kJ/m$^3$, respectively. The optimal $Na_2SiO_3$/NaOH ratio is found to be the same for all *E* values and L/FA ratios and is 80:20.
2. The optimal heat duration to manufacture the sludge-fly ash geopolymer is found to be identical for all sludge-FA geopolymer samples, irrespective of *E* and equal to 72 hours. However, the optimal heat temperature was dependent upon *E*; i.e., it was 75, 85 and 95 $^o$C for *E* = 2693.3, 1346.6 and 592.5 kJ/m$^3$, respectively. The maximum UCS at the optimal heat temperature and heat duration are 20, 18 and 16 MPa for *E* = 2693.3, 1346.6 and 592.5 kJ/m$^3$, respectively,

which meet the strength requirement as bearing masonry units.

3. SEM images clearly show the geopolymerization products on FA surface and in the pore space of the sludge-FA geopolymer heated at the optimal temperature. Overheating causes micro-cracks on the microstructure and results in the strength reduction.
4. This research will enable sludge traditionally destined for landfill to be used in a sustainable manner as an aggregate in geopolymer masonry units, which is significant in term of engineering, economical and environmental perspectives.

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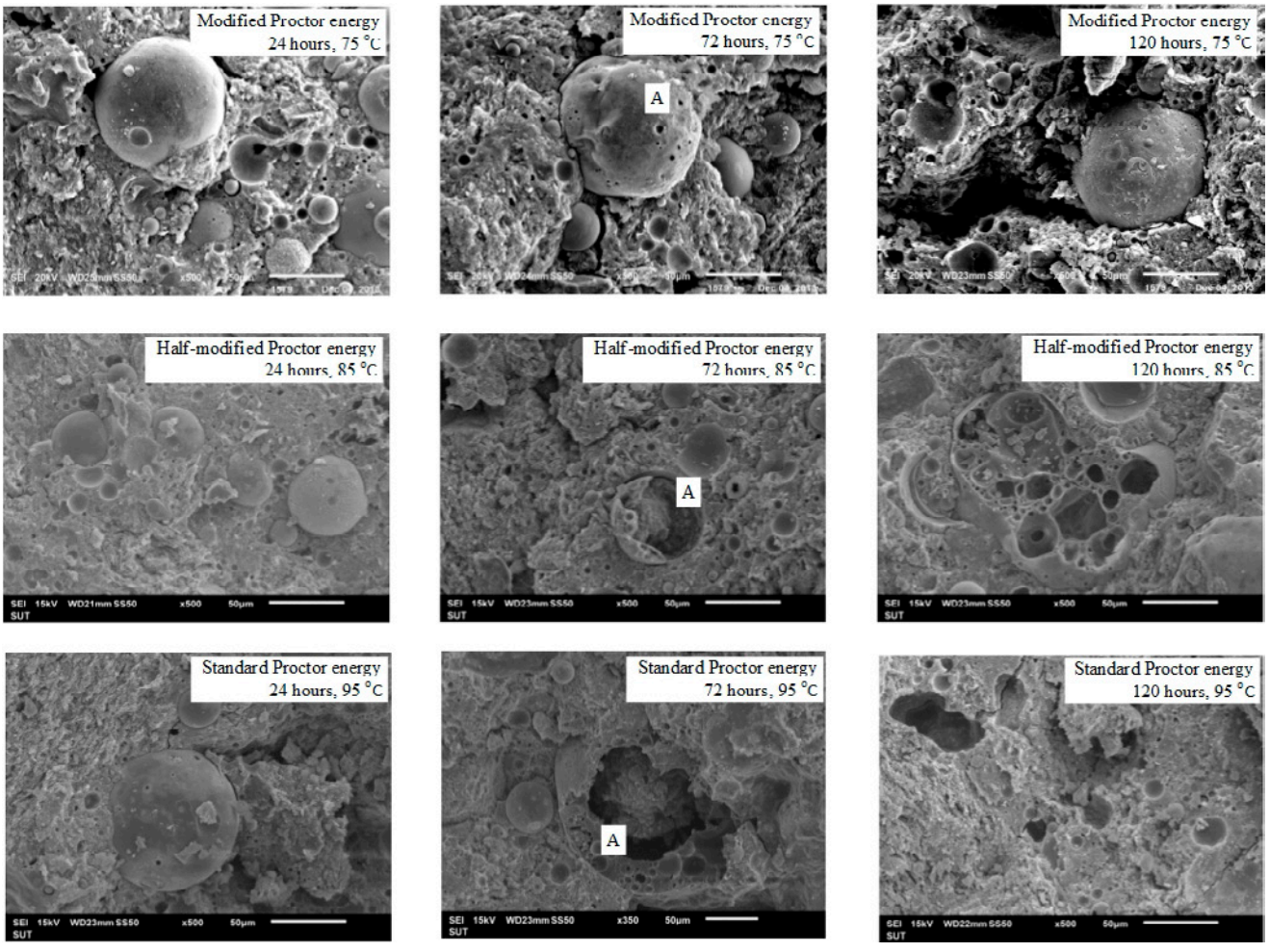


**Fig. 9.** SEM images of 7-day cured sludge-FA geopolymer at $Na_2SiO_3$/NaOH ratio of 80:20 and at various compression energies and heat durations.

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