Computers and Concrete, Vol. 9, No. 6 (2012) 427-437
DOI: http://dx.doi.org/10.12989/cac.2012.9.6.427

The effects of replacement fly ash with diatomite in geopolymer mortar

Theerawat Sinsiri*[1], Tanakorn Phoo-ngernkham[1], Vanchai Sata[2] and Prinya Chindaprasirt[2]

[1]*School of Civil Engineering, Institute of Engineering, Suranaree University of Technology, Nakhonratchasima 30000, Thailand*
[2]*Sustainable Infrastructure Research and Development Center, Dept. of Civil Engineering, Faculty of Engineering, Khon Kaen University, Khon Kaen 40002, Thailand*

(Received April 7, 2011, Revised July 29, 2011, Accepted August 22, 2011)

Abstract. This article presents the effect of replacement fly ash (FA) with diatomite (DE) on the properties of geopolymer mortars. DE was used to partially replace FA at the levels of 0, 60, 80 and 100% by weight of binder. Sodium silicate (Na_2SiO_3) and sodium hydroxide (NaOH) solutions were used as the liquid portion in the mixture in order to activate the geopolymerization. The NaOH concentrations of 15M, Na_2SiO_3/NaOH ratios of 1.5 by weight, and the alkaline liquid/binder (LB) ratios by weight of 0.40, 0.50, 0.60 and 0.70 were used. The curing at temperature of 75°C for 24 h was used to accelerate the geopolymerization. The flows of all fresh geopolymer mortars were tested. The compressive strengths and the stress-strain characteristics of the mortar at the age of 7 days, and the unit weights were also tested. The results revealed that the use of DE to replace part of FA as source material in making geopolymer mortars resulted in the increased in the workability, and strain capacity of mortar specimens and in the reductions in the unit weights and compressive strengths. The strain capacity of the mortar increased from 0.0028 to 0.0150 with the increase in the DE replacement levels from 0 to 100%. The mixes with 15M NaOH, Na_2SiO_3/NaOH of 1.5, LB ratio of 0.50, and using 75°C curing temperature showed 7 days compressive strengths 22.0-81.0 MPa which are in the range of normal to high strength mortars.

Keywords: geopolymer; diatomite; workability; compressive strength; strain capacity.

1. Introduction

Portland cement concrete is a mixture of Portland cement, water and aggregates. Nowadays, concrete is the most used construction material. However, the process of manufacturing of Portland cement consumes a large amount of energy and as a result releases a very large amount of green house gas to the atmosphere. The use of by-products or natural binders as cement replacement can, therefore, reduce the consumption of cement in concrete work. In the last decade, geopolymer binders have emerged as one of the possible alternative to cement binders for applications in concrete industry. Geopolymer is an inorganic binder material and can be produced by a polymeric reaction of alkali activating solution with silica and alumina in source material from geological origin or pozzolanic materials such as metakaolin, fly ash (FA) and rice husk ash (Davidovits 1991). The geopolymer mortar and concrete possess similar strength and appearance to those of normal

* Corresponding author, Ph.D., E-mail: sinsiri@sut.ac.th

Portland cement. Their mechanical properties, fire resistance and acid resistance are superior to those of normal Portland mortar and concrete (Palomo *et al.* 1999, Hardjito and Rangan 2005).

Diatomite (DE) or diatomaceous earth is a sedimentary deposit with origin from sedimentation of single cell seaweeds. It is light in weight due to high porosity and is relatively attractive in dark-yellow shade due to high content of ferrous compound and semi-crystalline siliceous phase (Owen and Utha-aroon 1999). The particle size is therefore quite fine with cellular surface consisting of micro-pores (Antonides 1999). DE has been used as a good source material for lightweight brick, heavy metals removal and absorbent agent in wastewater treatment process (Pimraksa and Chindaprasirt 2009, Elden *et al.* 2010). The deposit of DE or diatomaceous earth clay in Lampang province in the north of Thailand is quite large with estimation at more than 100 million tons. The mineral composition consists primarily of silica with some alumina and ferrous oxide (Sierra *et al.* 2010). It is a pozzolan and should, therefore, be suitable source material for making geopolymer.

The annual output of lignite FA from Mae Moh power station in the North of Thailand is around 3 million tons. This FA consists mainly of SiO_2, Al_2O_3, Fe_2O_3 and CaO and some impurities. The use of fly ash as pozzolan in concrete is constantly increasing because it improves the properties of concrete, namely workability, durability and long term strength in hardened concrete (Rukzon and Chindaprasirt 2008, Yeh 2008). In addition, it has also been shown to be suitable as a source material for making good geopolymer (Chindaprasirt *et al.* 2007).

This research aims to study the preparation of FA and DE geopolymer. The DE could be used for the adjustment of the silica content in the mixture. The knowledge of the use of high calcium lignite FA and silica rich DE in producing geopolymer would be beneficial to the understanding and to the future applications of the materials.

2. Experimental details

2.1 Materials

Lignite fly ash (FA) from Mae Moh power station and raw DE from Lumpang in the north of Thailand, sodium hydroxide (NaOH), and sodium silicate (Na_2SiO_3) with 15.32% Na_2O, 32.87% SiO_2 and 51.81% H_2O were the materials used. The raw DE was calcined at 800°C for 6 hours in order to improve its characteristics. (Yilmaz and Ediz 2008, Zuhua *et al.* 2009). The 6 hours calcination was used since the shorter period was found to be insufficient as the calcined products was still not properly burnt. For longer calcination, the formation of crystal could be detected. In addition, tap water and local river sand with specific gravity of 2.69 were used for making geopolymer mortar. In order to minimize the effect of the liquid absorption of fine aggregate, the sand in saturated surface condition was used.

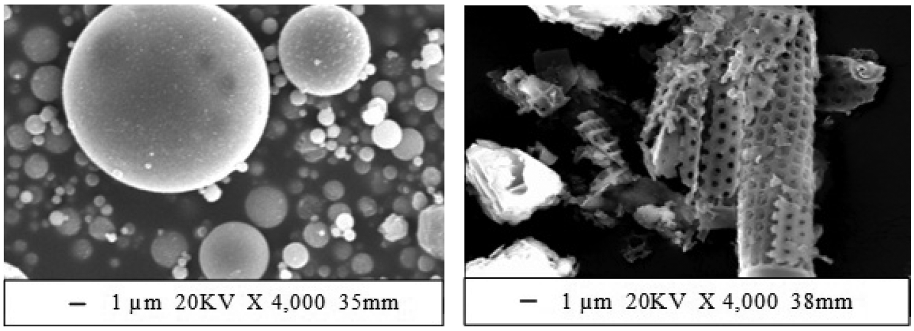
The chemical composition and physical properties of materials are given in Tables 1 and 2, respectively. The SiO_2+Al_2O_3+Fe_2O_3 content of the FA is 81.01% and the CaO content is high at

Table 1 Chemical composition of materials (by weight)

Materials	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	TiO	SO_3	LOI
FA	43.87	26.33	10.81	12.69	1.23	1.10	-	-	2.74	1.23
DE	59.30	10.00	18.50	1.20	0.46	1.98	0.20	0.23	0.02	8.10

Table 2 Physical properties of materials

Physical properties	FA	DE	Sand
Specific gravity	2.41	2.33	2.69
Bulk density (kg/m^3)	973	490	1625
Median particle size (μm)	17.6	18.3	-
Blaine fineness (cm^2/g)	4300	12600	-



(a) FA (b) DE

Fig. 1 The scanning electron micrographs of FA and DE

12.69% which is typical of the lignite FA. The DE consists of 59.30% SiO_2, 10.00% Al_2O_3 and 18.50% Fe_2O_3. FA has a mean particle size of 17.6 μm, a Blaine fineness of 4300 cm^2/g and a specific gravity of 2.41. DE has a mean particle size of 18.3 μm, a Blaine fineness of 12600 cm^2/g. Fig. 1 shows the scanning electron micrographs (SEM) of FA and DE. The FA particles are spherical and smooth in the surface while the DE particles consist of cellular porous particles with some small irregular shape plate-like particles.

2.2 Mix proportion and testing

All geopolymer mortars were made with sand to binder (FA+DE) ratio of 1.50. The FA100, FADE60, FADE80 and FADE100 mortars with corresponding FA:DE ratios of 100:0, 40:60, 20:80 and 0:100 were the mixes used. In addition, the effects of the *L/B* ratios on properties of geopolymer mortars were also studied.

The mixing was done in an air conditioned room at approximately 25°C to eliminate the possible effect of temperature variation. The NaOH and Na_2SiO_3 solutions were mixed before the start of the mixing. FA and DE were thoroughly mixed until the mixture was homogenous. Sand was incorporated into the blend of FA and DE and mixed for 1 minute. The prepared NaOH and Na_2SiO_3 solution was added and mixed for another 10 minutes. Right after the mixing, the flow values of fresh geopolymer mortar were the average of three samples and were tested in accordance with ASTM C 1437 (2003).

The fresh mortar was cast into 50 × 50 × 50 mm^3 cube moulds. The specimens were compacted in two layers and tamping as described in the ASTM C 109 (2003). The specimens were immediately wrapped with vinyl sheet to protect moisture loss and kept in the controlled room at 25°C. The specimens were then placed in the oven for heat curing at 75°C for 24 hours. After the heat curing, the specimens were put in the laboratory to cool down. They were demoulded the next day and kept in the control room. The compressive strengths of mortars were the average of three samples and were determined in accordance with ASTM C 109 (2003).

3. Results and discussions

3.1 Workability of geopolymer mortar

Fig. 2 shows the relationship between flow of geopolymer mortar and LB ratios at various replacement levels of DE. At low LB ratio, the geopolymer mortars were very stiff with low flow values between 2-16% for the LB ratio of 0.40 mortars. The workability of the mixes also increased with the increase the LB ratio. For example, the workable mortars with flow value of 85-126% were obtained with the LB ratio of 0.70. This result conforms to the previous research (Sathonsaowaphak *et al.* 2009) which explained that an increase in fluid medium content resulted in less particle interaction and increased the workability of the mixture. At the same LB ratio, the replacement FA with DE also increased the workability of mortar. At the LB ratio of 0.60, the flow of FA100, FADE60, FADE80 and FADE100 were 53, 82, 108 and 96%, respectively. The increasing of workability in mortar containing DE may be due to the low bulk density of DE as comparing with FA. The result also conformed to Torres and Garcia-Ruiz (2009) report that the use of lightweight pozzolan in cement mortar could be improved workability of mortar.

The empirical equations could be expressed for the workable geopolymer mortar with flow value (F) in terms of LB ratio with DE content of 0, 60, 80 and 100% as follows

For
$$\mathrm{DE} = 0\%,\quad F = 267.47(\mathrm{LB}) - 103.35,\quad (R^2 = 0.99) \tag{1}$$

$$\mathrm{DE} = 60\%,\quad F = 334.65(\mathrm{LB}) - 120.05,\quad (R^2 = 0.95) \tag{2}$$

$$\mathrm{DE} = 80\%,\quad F = 353.59(\mathrm{LB}) - 116.26,\quad (R^2 = 0.95) \tag{3}$$

$$\mathrm{DE} = 100\%,\quad F = 345.72(\mathrm{LB}) - 110.21,\quad (R^2 = 0.83) \tag{4}$$

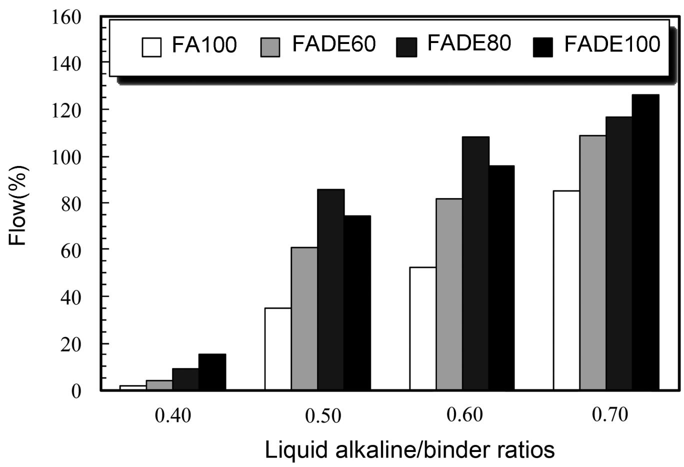
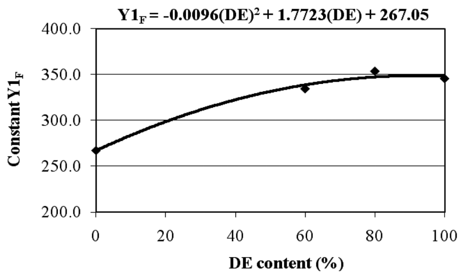
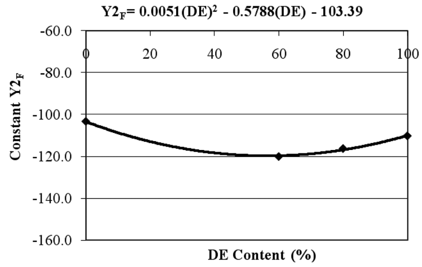


Fig. 2 Flows of geopolymer mortar with various LB ratios



(a) Constant $Y1_F$



(b) Constant $Y2_F$

Fig. 3 Relationship between constants $Y1_F$, $Y2_F$ and DE contents

The relationships of the flow value (F) and liquid alkaline/binder ratio (LB) in Eqs. (1)-(3) and (4) could be written as Eq. (5)

$$F = Y1_F(\mathrm{LB}) + Y2_F \tag{5}$$

Where

$$Y1_F = -0.0096(\mathrm{DE})^2 + 1.7723(\mathrm{DE}) + 267.05, \quad (R^2 = 0.99)$$

$$Y2_F = 0.0051(\mathrm{DE})^2 - 0.5788(\mathrm{DE}) - 103.39, \quad (R^2 = 0.99)$$

F = flow value of geopolymer mortar (%)
DE = amount of DE between 0 to 100%
LB = liquid alkaline/binder ratio between 0.40 to 0.70

The constants $Y1_F$ and $Y2_F$ were obtained by curve fitting of the results from Fig. 3 for DE content of 0, 60, 80 and 100%. The results of workability in term of flow from this equation were compared to the actual test results as shown in Fig. 4. The relationship in Eq. 5 is, therefore, useful to predict the flow of geopolymer mortar at various LB ratio and amount of replacement FA with DE. The non-linear behavior of flow of FADE80 and FADE100 mixes were due to the large

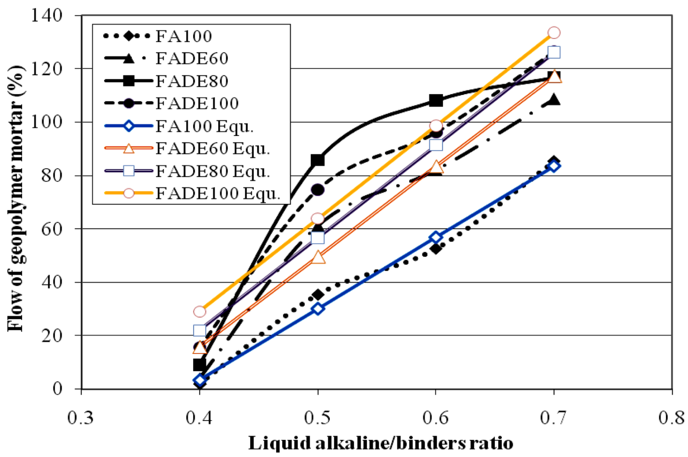


Fig. 4 Comparison of the flows of geopolymer mortar obtained from Eq. 5 and from experiment

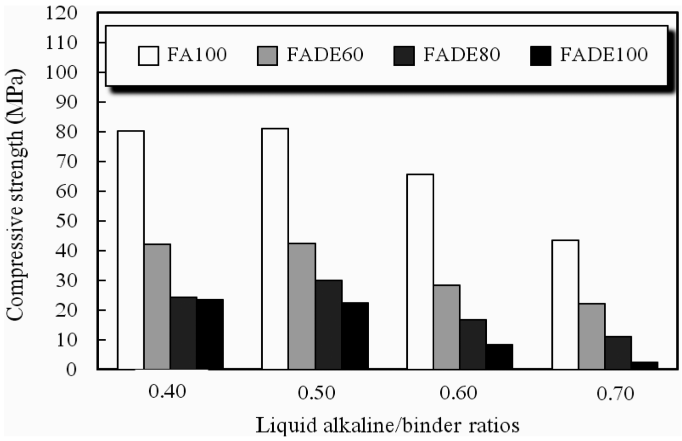


Fig. 5 Compressive strength of geopolymer mortar with various LB ratios

improvement of flow with high contents of DE of 80 and 100%. At 0.5 LB ratio, the large improvements of flow were obtained as a result of the large content of DE and an increase in the LB ratio. For higher LB ratios of 0.6 and 0.7, the small increases in the flow were obtained as they were approaching the obtainable maximum slump values.

3.2 The compressive strength of geopolymer mortar

The effect of LB ratios on 7 days compressive strength of geopolymer mortar at various DE contents is shown in Fig. 5. At the LB ratios of 0.40 and 0.50, the compressive strengths of the two series were almost the same. When the LB ratio was increased to 0.6 and 0.70, the compressive strengths of mortar decreased. For example, the 7 days compressive strength of FADE60 mortar at LB ratios of 0.40, 0.50, 0.60 and 0.70 were 42, 42, 28 and 22 MPa, respectively. Since, at high LB ratios may be due to excess of OH^- concentration in the mixture which decreased mortar compressive strength (Hardjito *et al.* 2008). In addition, Barbosa *et al.* (1999) indicated that the excess liquid solution could disrupt the polymerization process. The result also conformed to Sathonsaowaphak *et al.* (2009) report that the compressive strength of bottom ash geopolymer mortar seemed to decrease at high LB ratio (0.45-0.71).

The replacements of FA with DE in geopolymer mortar exhibited to decrease the compressive strength. At the LB ratio of 0.50, the 7 days compressive strengths of FA100, FADE60, FADE80 and FADE100 mortars were 81, 42, 30 and 22 MPa, respectively. This was due to the replacements of FA with DE caused to higher the SiO_2/Al_2O_3 ratios in the mixtures. From the chemical composition of FA and DE in this study, FA100, FADE60, FADE80 and FADE100 mortars had the SiO_2/Al_2O_3 ratio of 1.7, 4.2, 5.0 and 5.9, respectively. The suitable SiO_2/Al_2O_3 ratio for relatively high compressive strength geopolymer was around 1.9 (Duxson *et al.* 2005). In addition, many researchers (Duxson *et al.* 2005, Wongpa *et al.* 2010, Nazari *et al.* 2011) found that the SiO_2/Al_2O_3 ratio is the major parameter to control goepolymer matrix properties and at higher SiO_2/Al_2O_3 ratio resulted in low compressive strength.

The empirical equations could be expressed for the compressive strengths (C) of geopolymer mortars in terms of LB ratio and DE contents as follows

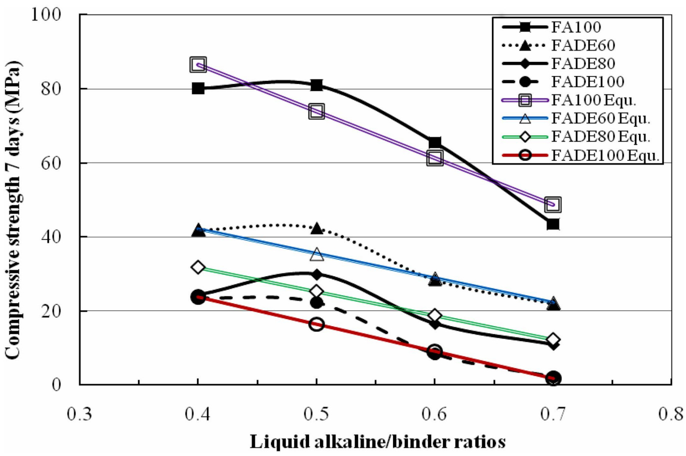


Fig. 6 Comparison of the 7 days compressive strengths of geopolymer mortars obtained from Eq. 6 and from experiment

$$C = Y1_C(\mathrm{LB}) + Y2_C \tag{6}$$

Where

$$Y1_C = -0.0117(\mathrm{DE})^2 + 1.6961(\mathrm{DE}) - 125.99, \quad (R^2 = 0.92)$$

$$Y2_C = 0.0075(\mathrm{DE})^2 - 1.5879(\mathrm{DE}) + 136.93, \quad (R^2 = 0.98)$$

C = compressive strength of geopolymer mortar (MPa)
DE = amount of DE between 0 to 100%
LB = liquid alkaline/binder ratio between 0.40 to 0.70

The 7 days compressive strengths from this equation were compared to the actual test result as shown in Fig. 6. The relationship in Eq. (6) is, therefore, useful to predict the 7 days compressive strength of geopolymer mortar at various LB ratios and amounts of replacement FA with DE.

3.3 The unit weight of geopolymer mortar

The unit weight FA and DE geopolymer mortar is shown in Fig. 7. It was found that the increasing LB ratio from 0.40 to 0.70 seemed to decrease the unit weight of mortar and the incorporation DE in the mixture decreased the unit weight of mortar. The FA100 (0% DE) mortars had unit weight range from 2180 to 2280 kg/m^3 and the use of 100% DE in geopolymer mortar (FADE100 mortar) reduced the unit weight to 1990 to 2100 kg/m^3. The reason is the low bulk density and high porosity of DE as compare to FA. Hence, at high LB ratio and the characteristic of DE can apply for lightweight geopolymer matrix.

The empirical equations of the unit weights geopolymer mortar (W) in terms of LB ratio and DE contents could be drawn as follows

$$W = Y1_W(\mathrm{LB}) + Y2_W \tag{7}$$

Where

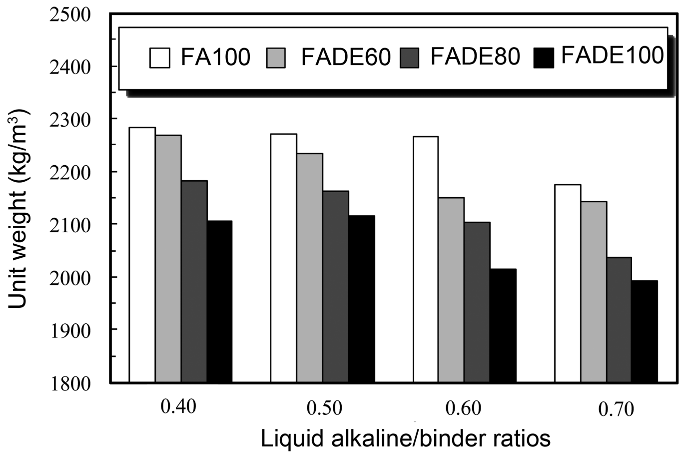


Fig. 7 Unit weights of geopolymer mortars with various of LB ratios

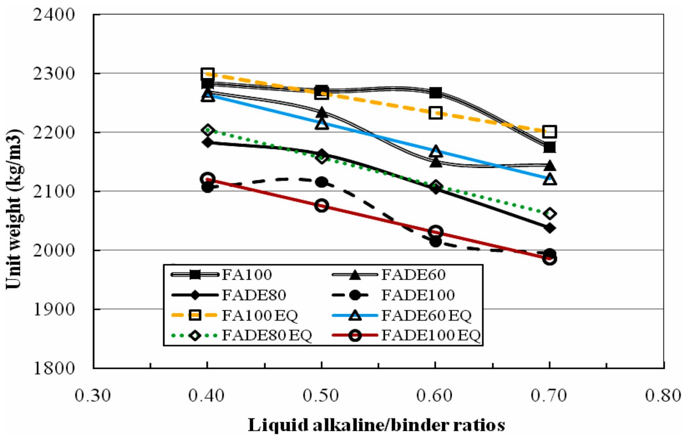


Fig. 8 Comparison the unit weight of geopolymer mortar obtained from Eq. (15) and from experiment

$$Y1_W = 0.0305(\mathrm{DE})^2 - 4.282(\mathrm{DE}) - 326.73, \quad (R^2 = 0.94)$$

$$Y2_W = -0.0419(\mathrm{DE})^2 + 2.9034(\mathrm{DE}) + 2429.3, \quad (R^2 = 1.00)$$

W = unit weight of geopolymer mortar (kg/m^3)
DE = amount of DE between 0 to 100%
LB = liquid alkaline/binder ratio between 0.40 to 0.70

Fig. 8 shows the unit weight of geopolymer mortar from Eq. (7) and the actual test results. It was found this equation is useful to predict the unit weight of geopolymer mortar at various LB ratios and amounts of replacement FA with DE.

3.4 The stress-strain characteristic of geopolymer mortar

The relationship between stress and strain of geopolymer mortars with the Na_2SiO_3/15 M NaOH ratio at 1.5 and the LB ratio of 0.50 at 7 days is shown in Fig. 9. The cord elasticity modulus was calculated as described in the ASTM C 469 (2003). The value of elastic modulus increased with the increase in compressive strength, which was similar to plain concrete. The replacement FA with DE reduced the compressive strength and the elastic modulus owing to an increase in the SiO_2/Al_2O_3 ratio (Pacheco-Torgal *et al.* 2008). However, the strain at peak stress (strain capacity) seemed to increase with DE content. Hence, the FA based geopolymer mortar mixes with DE exhibited a more deformable behavior. This reason is probably due to the high porosity and low bulk density of DE. The 7 days elastic modulus of FA100, FADE60, FADE80 and FADE100 mortars were 28.3, 12.0, 7.2 and 3.2 GPa with the strain at peak stress of 0.0028, 0.0047, 0.064 and 0.015, respectively.

Fig. 10 shows the relationship between modulus of elasticity and the compressive strength of geopolymer mortar. It was observed that the modulus of elasticity tended to increase linearly to the square root of compressive strength. The equation predicting this relationship can be drawn as follows

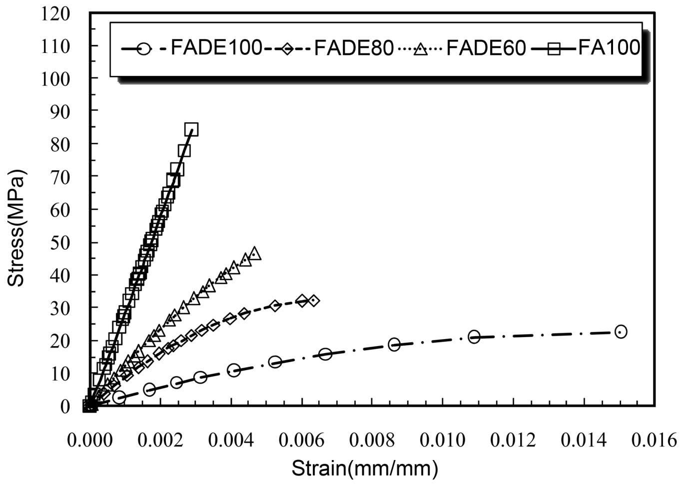


Fig. 9 Relationship between stress and strain of geopolymer mortar

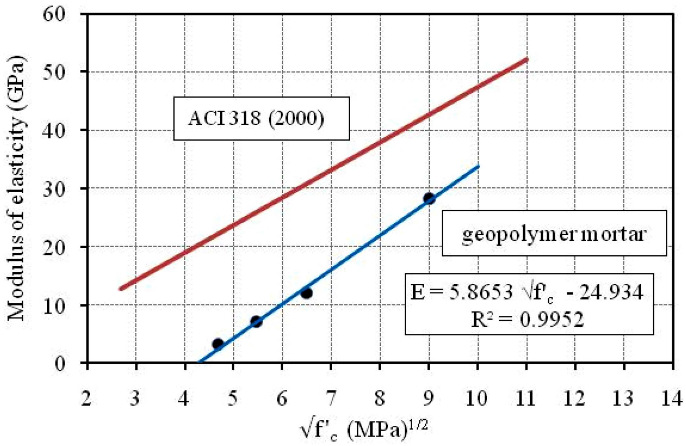


Fig. 10 Modulus of elasticity of geopolymer mortars

$$E = 5.8653\sqrt{f_c'} - 24.934 \tag{8}$$

Where

E = modulus of elasticity (GPa)

f_c' = compressive strength (MPa)

The values of modulus of elasticity in this study were lower than those predicted by ACI 318 (2000). The same trend of result was also reported by Wongpa *et al.* (2010) from the study of the compressive strength, modulus of elasticity and water permeability of inorganic polymer concrete. The increase in the strain capacity with the increase in the DE content is very useful in terms of the improvement in the cracking of this inorganic polymer matrix.

4. Conclusions

Based on the obtained data, it can be concluded that the DE could be used in conjunction with FA to produce good geopolymer mortars with improved workability, strain capacity and reduced unit weight of mortar. The use of DE to replace part of FA as source material in making geopolymer mortars resulted in an improvement in the workability of mortar. The compressive strength and modulus of elasticity of geopolymer mortar decreased with an increase in the DE content as a result of the increase in the SiO_2/Al_2O_3 ratio. The mixes with 15M NaOH, Na_2SiO_3/NaOH of 1.5, LB ratio of 0.50 and using 75°C curing temperature showed 7 days compressive strengths of 22.0-81.0 MPa. The strain capacity of the mortar, however, increased from 0.0028 to 0.0150 with the increase in the DE replacement. In addition, the incorporation DE in the mixture decreased the unit weight of mortar. The improvement in the workability, strain capacity and unit weight with the incorporation of DE can be used to advantageous in designing the geopolymer mortars using these based materials.

Acknowledgements

The authors would like to acknowledge the financial support by the Higher Education Research Promotion and National Research University Project of Thailand, Office of the Higher Education Commission, through the Advanced Functional Materials Cluster of Khon Kaen University. Thanks are also extended to the Thailand Research Fund (TRF) under the TRF New Researcher Scholar, and grant No. MRG5280178, the National Research Council of Thailand.

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