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High calcium fly ash geopolymer containing diatomite as additive

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This paper studies the influence of the addition of diatomite on the properties of high calcium fly ash geopolymer pastes. Diatomite is calcined at 800°C and used to partially replace fly ash at the rate of 0, 10, 20, 30 and 40% by weight of binder. Sodium silicate (Na_2SiO_3), sodium hydroxide (NaOH) solution and curing at temperature of 60°C for 24 h are used to activate the geopolymerization. The NaOH concentration of 10 M and Na_2SiO_3 /NaOH ratio of 2.0 by weight are used for all mixes. The results revealed that the use of diatomite to replace part of fly ash delayed the setting time of fresh geopolymer paste and also resulted in the reduction of density of hardened paste which is highly desirable for lightweight structure member. However, the strength and modulus of elasticity of the paste are reduced. Another positive effect is the increase in the strain capacity of the paste. The increase in strain capacity is significant and reflects in the ability of the paste to withstand increased strain before cracking.

Keywords: Geopolymer, High calcium fly ash, Diatomite, Compressive strength, Unit weight, Strain capacity

The production of Portland cement (OPC) results in the emission of a large amount of carbon dioxide (CO_2) into the atmosphere¹. To solve this problem, industrial and agricultural by-products such as fly ash, rice husk ash, and palm oil fuel ash are used as pozzolans to replace part of OPC^{2-5} . These materials also improve the properties of concrete namely durability and long-term strength of concrete^{6,7}.

In the last decade, geopolymer binder emerged as an environmental friendly alternative to normal cement binders for applications in concrete industry. It is made from silica and alumina rich source materials such as fly ash and calcined kaolin. The geopolymeric reaction relies on the activation with alkali solutions and temperature curing at 40-75° C^8 . The geopolymer possesses similarly strength and appearance to normal Portland cement. Their mechanical properties, fire resistance and acid resistance are superior to those of normal Portland cement mortar and concrete⁹.

One of the important properties of the geopolymeric material is setting time. The fresh geopolymer paste usually has a short setting time especially when the calcium content of the system is

high^{10,11}. The other important property of the cementitious products is the ability to withstand large strain and thus reduce the chance of crack formation. This property is related to the amount of strain which the material can withstand before it fractures. For fly ash geopolymer paste, the strain at peak stress was increased with the addition of diatomite¹². The increase in the replacement level of diatomite also produces concrete with reduced unit weight¹²⁻¹⁴ which is desirable.

Fly ash is a by-product from coal burning in the power plant. In Thailand, the major source of fly ash is from a lignite coal and consists mainly of silicon dioxide (SiO₂), aluminum trioxide (Al₂O₃) and calcium oxide (CaO). It is a suitable source material for making good geopolymer^{11,15}. The high calcium content of fly ash leads to the formation of calcium silicate hydrate which co-exists with the aluminosilicate geopolymer products^{8,16} and improves the mechanical properties of the final product¹⁷.

Diatomite (DE) or diatomaceous earth clay is a sedimentary deposit with origin from sedimentation of single cell seaweeds. The deposit in Lampang province in northern Thailand is quite large with estimation at more than 100 million tons. It consists mainly of silica with some alumina and ferrous

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oxide¹⁸. Many researchers have considered using DE in cementitious binder which is desirable. DE is light in weight due to high porosity¹⁹, therefore, the DE can be used in order to reduce the unit weight of the structure, lightweight construction materials in the form of lightweight concrete block or wall lightweight have been developed¹³. Moreover, Pimraksa *et al.*¹⁴ studied the lightweight geopolymer made of highly porous siliceous materials, viz., diatomite and rice husk ash and found that the DE appears to be a good candidate for producing lightweight geopolymer material. Consequently, the DE could be a suitable material to adjust the silica content in the mix for making good fly ash geopolymer with reduced unit weight.

This research aims to study the influence of incorporation of DE on the properties of geopolymer paste. It is expected that the DE can be used for the adjustment of the silica and alumina content and the setting time of fresh geopolymer paste. In the hardened state, the incorporation of DE should increase the strain at peak stress and lower the weight of the products. The obtained knowledge should be very beneficial to the understanding and to the future applications of the materials.

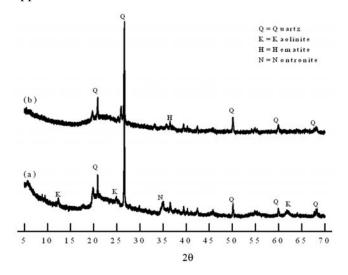


Fig. 1 – XRD patterns of DE (a) uncalcined DE and (b) calcination at $800^{\circ}C$

Experimental Procedure

Materials

The materials consisted of fly ash (FA) from Mae Moh Power Plant and diatomite (DE) from Lampang Province in Northern Thailand. The liquid portions in the mixture were 10 M sodium hydroxide (NaOH), and sodium silicate (Na₂SiO₃) with 13.89% Na₂O, 32.15% SiO₂ and 46.04% H₂O. The raw DE was calcined at 800°C for 6 h in order to improve its characteristics^{20,21}. The calcination at 800°C resulted in some hematite and additional quartz and the disappearance of the kaolinite and nontronite peaks. This corresponded to the dehydroxylation of kaolinite when clay mineral (kaolinite) was calcined and thus became active²² as shown in Fig. 1. The six 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 a large amount of crystal could be detected.

The chemical compositions of the FA and DE are shown in Table 1. The sum of $SiO_2+Al_2O_3+Fe_2O_3$ for FA and DE were 57.92% and 87.80%, respectively, while the CaO content of FA is high at 25.79%, The high calcium content suggested that it belonged to the class C fly ash whereas DE could be classified as Class N pozzolan as specified by ASTM C618²³. The median particle size, Blaine fineness, and specific gravity of FA were 8.5 µm, 4300 cm²/g and 2.61, respectively, and those of DE were 18.3 µm, 12600 cm²/g and 2.33, respectively as shown in Table 2.

Mix proportion

The mix proportions of geopolymer pastes are given in Table 3. The FA was partially replaced with DE at the rates of 0, 10, 20, 30, and 40% by weight of binder with addition super plasticizer (SP) was 0.75-3.00 of weight binder to make homogeneous pastes. Thus, the range of water to cement (W/C) ratio or liquid to binder (L/B) ratio was 0.6-0.77. The increase in the W/C or L/B ratio was due to the increase in the water requirement of the DE to produce paste with similar workability. The constant

Table 1 – Chemical composition of materials (by weight)										
Materials	SiO_2	Al_2O_3	Fe ₂ O ₃	CaO	MgO	K ₂ O	Na ₂ O	TiO	SO ₃	LOI
FA	29.32	12.96	15.64	25.79	2.94	2.93	2.83	-	7.29	0.30
DE	59.30	10.00	18.50	1.20	0.46	1.98	0.20	0.23	0.02	8.10

			Table 2	- Physical propert	ies of materials			
Materials	Specific gravity		Bulk density (kg/m ³)		Median particle size, d_{50} (µm)		Blaine fineness (cm ² /g)	
FA		2.61	1055		8.5		4300	
DE	2.33		490		18.3		12600	
	Table 3 – Mix proportion of geopolymer pastes							
Mix No.	Symbol	FA (%)	DE (%)	Liquid/binder ratios	Super plasticizer (%)	SiO ₂ /Al ₂ O ratios	Na ₂ O/SiO ₂ ratios	
1	Control	100	0	0.60	0	3.25	1.01	
2	FA10DE	90	10	0.64	0.75	3.53	0.98	
3	FA20DE	80	20	0.68	1.50	3.80	0.94	
4	FA30DE	70	30	0.73	2.25	4.08	0.89	
5	FA40DE	60	40	0.77	3.00	4.35	0.82	

of the 10 M NaOH concentration and the Na_2SiO_3 to NaOH ratio of 2.0 were used for all mixes based on the earlier research²⁴. The SiO₂/Al₂O₃ ratios ranged from 3.25-4.35 and were within the suggested range of 4.0 by Davidovits²⁵.

For mixing of pastes, the NaOH and Na_2SiO_3 solutions were mixed together prior to the start of mixing. In order to obtain a good homogeneity, the FA and DE were mixed together until a uniform blend was obtained which took approximately one minute. The NaOH and Na_2SiO_3 solutions were then added and the mixture was mixed for another 5 min.

Determination of setting time

After the mixing, the setting time of the geopolymer paste were tested as described in ASTM C 191^{26} using the Vicat needle. The reported results were the average of two samples which were within the range required by the ASTM C191²⁶ for the single-operator.

Determination of stress-strain curve, modulus of elasticity and compressive strength

The fresh paste was placed in $50 \times 50 \times 50$ mm cube moulds in accordance with the ASTM C 109^{27} and in the 50 mm diameter and 100 mm height cylindrical moulds. The $50 \times 50 \times 50$ mm cube samples were used for the compressive strength test and the 50×100 mm cylinder samples were used for the determination of stress-strain curve and modulus of elasticity. The specimens were immediately wrapped with vinyl sheet to protect moisture loss and were put in 23° C controlled room for a delay period of one hour. The specimens were then place in the oven for heat curing at 60° C for 24 h. The specimens were demoulded at the age of approximately 25 h and kept in the 23° C Table 4 – Initial and final setting time of geopolymer pastes

		Setting time (min)		
Mix no.	Symbol	Initial	Final	
1	Control	30	58	
2	FA10DE	45	102	
3	FA20DE	68	119	
4	FA30DE	96	177	
5	FA40DE	182	274	

controlled room. The specimens were tested to determine the stress-strain curve, modulus of elasticity and compressive strength at the ages of 1, 7, 28, and 90 days. The specimens were placed in the compression machine and the compressive strength test was performed on the specimen according to ASTM C 109^{27} with the load and the displacement were continuously monitored. The chord modulus of geopolymer paste was calculated as described in ASTM C 469^{28} . The peak stress and the strain as peak stress were also recorded. The reported results were the average of three samples.

Determination of unit weight

The unit weight of geopolymer paste was determined as described in ASTM C 138^{29} . The unit weight was measured at the ages of 1, 7, 28, and 90 days using the compressive strength specimens. The reported results were the average of three samples.

Results and Discussion

Setting time of geopolymer paste

Table 4 shows the setting time of geopolymer pastes with various levels of DE replacement. The increase in DE content obviously increased the setting time of paste. The presence of calcium caused a rapid setting of the geopolymer paste³⁰. The replacement of high calcium FA with DE resulted in a reduction in the calcium content of the mixture and thus delayed the setting time. Furthermore, in order to keep the workability of the mixes similar, the paste content of the mixes needed to be increased with the increase in the DE content due to the highly porous characteristics of DE¹³. The increase in the liquid portion of the mix reduced the concentration of the leached ions of silica, alumina, and calcium. This also contributed to the delay of the setting time of paste. The DE was rich in SiO₂ and thus the SiO₂/Al₂O₃ ratio increased with the increase in the DE content which affected the reaction degrees of the geopolymer³¹. The high SiO₂/Al₂O₃ ratios of geopolymer generally delayed the setting time 31 .

The compressive strength of geopolymer paste

The compressive strengths and normalized compressive strength of geopolymer pastes at various DE contents are given in Table 5. The compressive strengths of pastes increased with time, but decreased with increasing replacement level of DE. The increase in strength with time was due to the addition of DE enriched the Si in the matrix and allowed strong Si-O-Si bonds to form³². In addition, the high calcium content of FA leads to the formation of additional C–S–H which coexists with the products^{33,34}. aluminosilicate geopolymer The coexistence of C-S-H phase with the geopolymeric gel had been shown to improve the mechanical properties of the final product¹⁷ similar increase in the strength with time was reported by several researches^{32,35}. However, for 28 to 90 days, the strength marginal increased as also observed by the other researchers Pacheco-Torgal et al.³⁶ and Chindaprasirt *et al.*³⁷. The small increase is due to the accelerated reaction caused by the heat curing similar that of the Portland cement system¹¹. The reduction in FA with the corresponding increase in the DE resulted in a high SiO_2/Al_2O_3 ratio of the mixture and thus resulted in the low compressive strength^{38,39}. The suitable SiO_2/Al_2O_3 ratio for relatively high compressive strength geopolymer cement was approximately 3.00^{40} . Furthermore, the reduction in the strength was also due in part to the reduction in the amount of Na₂O as described by the Na₂O/SiO₂ (or Na₂O/Al₂O₃ ratios) as it indicated the amount of OH⁻ in the mixture. The results confirmed with earlier research¹⁴ that the decrease in the Na₂O/SiO₂ ratios reduced the strength of the geopolymer paste.

From Fig. 2, the normalized compressive strength of pastes due to the replacement of DE is in the exponential form and the equation to predict the normalized compressive strength of pastes due to the replacement of DE is proposed as Eq. (1). The relations of the strength with DE replacement level were best fitted with the exponential decay function as the increase in the DE replacement level further reduced the strength of the paste. The normalized compressive strengths of pastes from the experimental data are presented in Eqs (2-5).

$$C_p = k \exp^{-a(DE)} \qquad \dots (1)$$

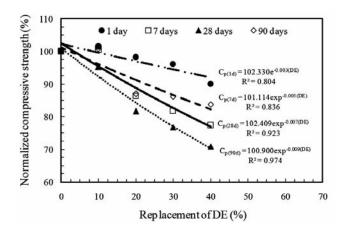


Fig. 2 – Relationship between normalized compressive strength of pastes and replacement of DE

Table 5 - Compressive strength of geopolymer pastes

		Compressive strength (MPa)-normalized (%)					
Mix no.	Symbol	1 day	7 days	28 days	90 days		
1	Control	32.5-(100)	42.0- (100)	48.2 -(100)	48.8- (100)		
2	FA10DE	33.0-(101)	42.2-(101)	45.9-(95)	49.2 -(101)		
3	FA20DE	31.9-(98)	36.2 -(86)	39.4- (82)	42.4-(87)		
4	FA30DE	31.2-(96)	34.3- (82)	37.0-(77)	42.0-(86)		
5	FA40DE	29.3-(90)	32.5-(77)	34.1-(77)	40.8-(84)		

For

1-day,
$$C_{p(1d)} = 102.332 \exp^{-0.003DE}$$
 ... (2)

7-days,
$$C_{p(7d)} = 101.114 \exp^{-0.005DE}$$
 ... (3)

28-days,
$$C_{p(28d)} = 102.409 \exp^{-0.007 DE}$$
 ... (4)

90-days,
$$C_{p(90d)} = 100.900 \exp^{-0.009 DE}$$
 ... (5)

Where C_p is normalized compressive strength of paste due to replacement of DE (%), *k*,*a* are compressive strength constants of pastes, and *DE* is percentage of replacement DE (%).

The unit weight of geopolymer paste

The unit weight and normalized unit weight of FA and DE geopolymer paste are given in Table 6. The results indicated that the increasing DE content tended to decrease the unit weight. The control paste had unit weight range between 2472-2527 kg/m³ whereas the geopolymer pastes with 10-40% DE content had the reduced unit weights of 2140-2383 kg/m³. The unit weight of paste incorporating DE resulted from the low bulk density and high porosity of DE as compare with that of FA, therefore, can be apply for lightweight geopolymer.

From Fig. 3, the normalized unit weight of pastes due to the replacement of DE is in linear form and the equation to predict the normalized unit weight of pastes due to the replacement of DE is proposed as Eq. (6). The relations of the density with DE replacement level were fitted with the linear function as the increase in the DE replacement level should linearly decrease the density of the paste. The normalized unit weights of pastes from the experimental data are presented in Eqs (7-10).

$$W_p = a(DE) + k \qquad \dots (6)$$

For

1-day, $W_{p(1d)} = -0.328(DE) + 99.194$... (7)

7-days, $W_{p(7d)} = -0.313(DE) + 98.149$... (8)

28-days,
$$W_{p(28d)} = -0.356(DE) + 98.290$$
 ... (9)

90-days,
$$W_{p(90d)} = -0.335(DE) + 98.268$$
 ... (10)

where W_p is normalized unit weight of paste due to replacement of DE (%), k,a are unit weight constants of pastes, and DE is percentage of replacement DE (%).

The modulus of elasticity and strain capacity of geopolymer paste

The modulus of elasticity and strain of geopolymer pastes are given in Table 7. The increasing DE content obviously decreased the modulus of elasticity whereas the strains at peak stress (strain capacity) increased. The elastic modulus at 90 days of the control, FA10DE, FA20DE, FA30DE, and FA40DE pastes were 15.9, 15.4, 13.8, 12.2, and 12.0 GPa, respectively which were comparable

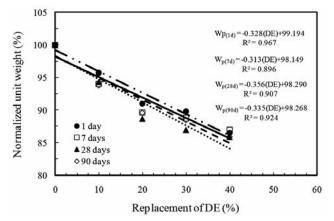


Fig. 3 – Relationship between normalized unit weight of geopolymer paste and replacement of DE

Table 6 – Unit	weight of geo	polymer pastes

	Unit weight (kg/m ³)-normalized (%)						
Mix no.	Symbol	1 day	7 days	28 days	90 days		
1	Control	2472-(100)	2521-(100)	2527-(100)	2518-(100)		
2	FA10DE	2367- (96)	2371-(94)	2383-(94)	2362-(94)		
3	FA20DE	2251-(91)	2259-(90)	2241-(89)	2256-(90)		
4	FA30DE	2221 -(90)	2238-(89)	2197-(87)	2228- (88)		
5	FA40DE	2140-(87)	2193-(87)	2170- (86)	2163- (86)		

to those of normal strength Portland cement pastes of 12.5-17.5 GPa^{41,42}. The strain at peak stress of the control paste at the age of 28 days was 0.00263 mm/mm and increased to 0.00374 mm/mm with the DE replacement level of 40%. The increase in strain at peak stress is significant and reflects in the ability of the sample to withstand increased strain before cracking. The reduction in compressive strength and modulus of elasticity and the increase in the strain at peak stress (strain capacity) were due to the increases in the SiO₂/Al₂O₃ ratios⁴³ leading to a more deformable behavior. In addition, the incorporation of DE of high porosity and low bulk density also contributed to these behavior^{13,14}.

The relationship between modulus of elasticity and compressive strength of geopolymer paste is shown in Fig. 4. The modulus of elasticity tended to increase linearly with the square root of compressive strength as suggested by ACI 318 (2002). The relationship could be predicted in the form shown in Eq. (11):

$$E = 4.458\sqrt{f_c'} - 15.554 \qquad \dots (11)$$

where E is modulus of elasticity (GPa), and f_c' is compressive strength (MPa).

The R^2 was 0.941 indicating the goodness of fit. Furthermore, the values of modulus of elasticity of the paste in this study were similar to those of the Portland cement paste⁴⁴ and about half of those given by ACI 318 for Portland cement concrete⁴⁵ with similar strength between 38.5 to 52.2 MPa as shown in Fig. 4. The results also suggest that the increase in DE content is very useful in terms of the improvement in the cracking strain of this inorganic polymer matrix.

Relationship between compressive strength and unit weight of geopolymer paste

The graph between the compressive strength and unit weight of geopolymer pastes is shown in Fig. 5. The compressive strength of paste increased exponentially with increasing unit weight and confirmed to the previous reported results^{14,46}.

Figure 5 shows that the empirical equations could relate the compressive strength and unit weight of geopolymer pastes (C) with various DE replacement levels of 0, 10, 20, and 30% as shown in Eqs (12-15).

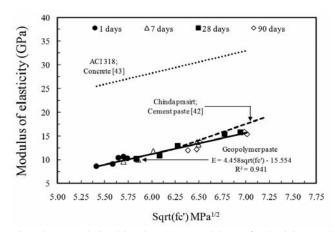


Fig. 4 – Relationship between modulus of elasticity and compressive strength of geopolymer paste

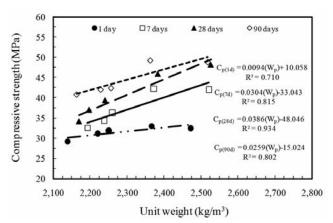


Fig. 5 – Relationship between compressive strength and unit weight of geopolymer paste

	Table 7 – Mo	odulus of elasticity and	strain capacity of geopo	olymer pastes					
	_	Capacity of modulus of elasticity and strain capacity at peak stress							
Туре	Symbol	1 day	7 days	28 days	90 days				
	Control	10.6	13.7	15.7	15.9				
	FA10DE	10.3	13.2	15.4	15.4				
E (GPa)	FA20DE	10.4	11.8	12.8	13.8				
. ,	FA30DE	9.1	10.0	10.8	12.2				
	FA40DE	8.6	9.6	10.1	12.0				
	Control	0.00390	0.00302	0.00263	0.00260				
	FA10DE	0.00407	0.00318	0.00292	0.00273				
$\boldsymbol{\mathcal{E}}_{n}$ (mm/mm)	FA20DE	0.00412	0.00330	0.00304	0.00282				
p <	FA30DE	0.00416	0.00360	0.00334	0.00294				
	FA40DE	0.00437	0.00393	0.00374	0.00313				

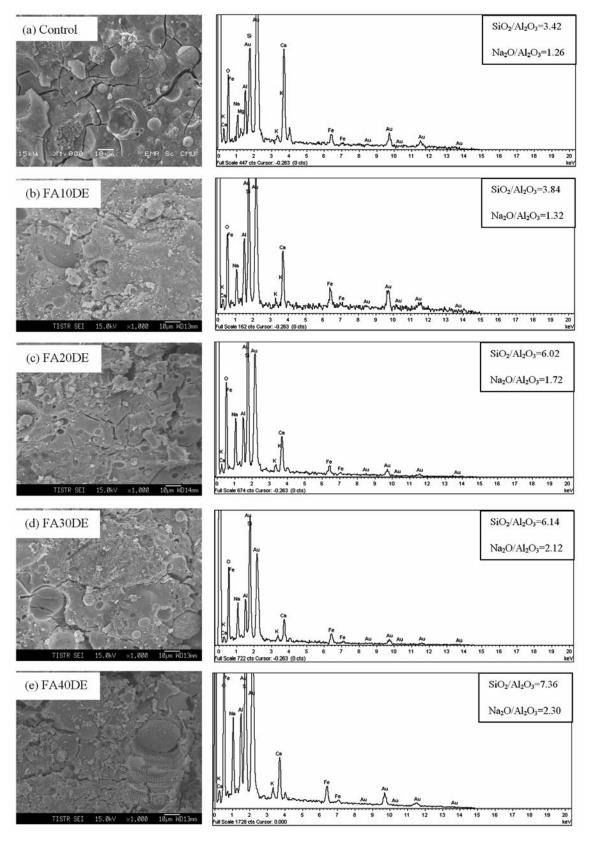


Fig. 6 – SEM/EDX of geopolymer paste

For

1 day,
$$C_{(1d)} = 0.0094W + 10.058$$
 ... (12)

7 days, $C_{(7d)} = 0.0304W - 33.043$... (13)

28 days,
$$C_{(28d)} = 0.0386W - 48.046$$
 ... (14)

90 days,
$$C_{(90d)} = 0.0259W - 15.024$$
 ... (15)

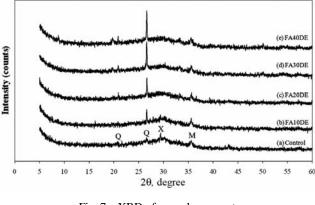
Where C is compressive strength (MPa), and W is unit weight (kg/m^3) .

The increase in replacement level of DE reduced the unit weight of the paste as a result of the low unit weight of DE. The low unit weight could be used to advantages in the reduction of the dead load of the construction member. The results also suggested that replacement level of DE can be applied for lightweight structure member which has density and strength of 1760 kg/m³ and 17.24 MPa as described in ACI 213R-87⁴⁷.

SEM/EDX analysis of geopolymer paste

The SEM photos of geopolymer pastes are shown in Fig. 6. The control paste showed dense more than the mix containing DE, and the control paste contained a larger number of non-reacted and/or partially reacted fly ash particles embedded in a continuous matrix (Fig. 6a). The addition DE showed some void and un-reacted DE¹³ could be observed in the sample of FA10DE, FA20FA, FA30DE, and FA40DE (Fig. 6b-6d). For the FA40DE paste, more amounts of the DE particles were observed and the matrix appeared less dense than that of the other mix. Although, the addition DE approximately 10% showed good replacement could be similarly strength and reduction unit weight when compare with the control paste.

The EDX analysis of geopolymer pastes showed major element of Si, Al and Na. The increase DE content increasing the SiO₂/Al₂O₃ and Na₂O/Al₂O₃ ratios. The physical and mechanical properties of geopolymer were a function of SiO₂/Al₂O₃ and Na₂O/Al₂O₃ ratios²⁴. The geopolymer paste with the SiO₂/Al₂O₃ ratios of 3.42 and 3.84, the Na₂O/Al₂O₃ ratios of 1.26 and 1.32 (as showed in Fig. 6a and 6b) confirmed with reported of SEM photos which the presence of continuous and dense mass of strong



Q=Quartz (SiO₂), X=CSH (Ca_{1.5}SiO_{3.5}.xH₂O), M=Magnesioferrite (MgFe₂O₄)

Fig. 7 – XRD of geopolymer paste

geopolymer. For high the Na_2O/Al_2O_3 ratio with increasing DE content, the silicate phases were more susceptible to washer dissolution¹⁴.

XRD analysis of geopolymer paste

The XRD patterns of geopolymer pastes are shown in Fig. 7. The control paste was amorphous phases as indicated by broad hump at the region of $25-35^{\circ}2\theta$, the main phases containing quartz (SiO₂), calcium silicate hydrate (Ca_{1.5}SiO_{3.5} xH₂O) and magnesioferrite (MgFe₂O₄). For addition of DE, this could be obviously increase quartz (SiO₂) and decrease calcium silicate hydrate (Ca_{1.5}SiO_{3.5}.xH₂O). Although the FA10DE seemed to a similar the control paste, this led to the overall similar strength of geopolymer products.

Conclusions

Based on the results of this study, the following conclusions could be drawn. The use of DE to replace part of high calcium FA in making geopolymer paste is beneficial as it delays the setting time, reduces the unit weight and increases the strain capacity of the fly ash geopolymer paste. The increase in the setting time is due to a reduction in the total calcium content and increase in the SiO₂/Al₂O₃ ratio of the system, while the reduction in the unit weight is due to the low bulk density and high porosity of DE. The increase in the strain capacity is due to the high SiO₂ content of the mixture which results in the more deformable material more than failure behavior of brittle material, this behavior can be appled for any structure member to improvement cracking with DE content. In addition, the addition DE were approximately 10% showed similar strength and reduction unit weight compare with control paste. It should be noted that the use of DE although beneficial in terms of lower the unit weight and increasing the strain capacity of the paste, the cost and the increase in the energy has to be considered.

Acknowledgements

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