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Lightweight geopolymeric materials from highly siliceous materials

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Abstract

The syntheses of lightweight geopolymeric materials highly siliceous materials viz. diatomaceous earth (DE) and rice husk ash (RHA) using SiO_2/Al_2O_3 ratios of 13.0-33.5 and Na_2O/Al_2O_3 ratios of 0.7-3.0 were studied. The effects of fineness and calcination temperature of DE, concentrations of NaOH and KOH, DE to RHA ratio, curing temperature and time on the mechanical properties and microstructures of the geopolymer pastes were studied. The optimum calcination temperature of DE was 800 °C. The increases in fineness of DE and in alkali concentration resulted in an increase in compressive strength of geopolymer paste. Geopolymer pastes activated with NaOH gave higher compressive strengths than those with KOH. The optimum curing temperature and time were 75 °C and 5 days. The lightweight geopolymeric material with bulk density of 880 kg/m^3 and compressive strength of 1.5 MPa was obtained. The SiO_2/Al_2O_3 and Na_2O/Al_2O_3 ratios of the material were 13 of 1.0, respectively. The incorporation of 40% RHA that changed SiO_2/Al_2O_3 and Na_2O/Al_2O_3 ratios to 22.5 and 1.7 enhanced the compressive strength of geopolymer paste to 2.4 MPa with only a slight increase of bulk density to 1,010 kg/m^3.

Keyword: Diatomaceous earth; Rice Husk Ash; Geopolymer; Lightweight materials

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1. Introduction

Manufacture of Portland cement is energy intensive and emits a large amount of carbon dioxide of approximately 7% of global emission [1]. The process also produces pollution in the forms of dust, noise and vibration with operating machinery and during blasting in quarries. Efforts, therefore, have been made to develop alternative cementitious materials to reduce the mentioned problems. One of the promising findings is to develop a geopolymeric material with low CO_2 emission and low energy requirement. The geopolymeric material is produced from the dissolution of alumino-silicate oxides and the polymerization reaction of those dissolved species in the presence of alkali ions for charge balancing in framework cavities nearby AlO_4 location. The reaction forms SiO_4 and AlO_4, tetrahedral frameworks linked by shared oxygens as polysialates or polysialate-siloxo or polysialate–disiloxo depending on the SiO_2/Al_2O_3 ratio in the system [2]. The connection of the tetrahedral frameworks is performed by covalent bonds in the long range. Geopolymer structure is therefore perceived as dense amorphous and/or semi-crystalline 3-D alumino-silicate microstructure.

Nowadays, lightweight construction materials are used to reduce the weight of building structures and improve thermal insulation of buildings. This study, therefore, focused on the production of lightweight geopolymer. Suitable lightweight source materials containing silica and/or alumina and are abundant in Thailand are diatomaceous earth (DE) and rice husk ash (RHA). DE is a sedimentary rock consisting principally of highly porous skeletons of diatom, in other word, a loosely packed mineral. The porous silica structure gives DE useful characteristics such as unique particulate structure, low bulk density, high absorptive capacity and high surface area [3]. The deposit of DE in Lampang province in the north is quite large with estimation of more than 100 million tons. The mineral composition consists primarily of silica with some clay minerals viz. montmorillonite, kaolinite and muscovite [4]. These clay minerals render DE as a suitable source for making geopolymer. Rice husk ash (RHA) is considered useful to help increase the dissolved SiO_4 species during dissolution and for subsequent polymerization stages [X: S. Detphan, P. Chindaprasirt, Preparation of fly ash and rice husk ash geopolymer, International Journal of Minerals, Metallurgy and Materials,.......].

It is still not certain whether 3D built-up structures of geopolymer made of highly porous silica are impeded due to its characteristic long ranged bonding of inorganic polymer chains. This research, therefore, aims to study reduction of unit weight, developments of strength and microstructure after the chemical reaction of polymerization. Thermal conductivity is also investigated. The knowledge of the production of DE lightweight geopolymer should be beneficial to the understanding of the mechanism and to develop the lightweight materials.

2. Materials and methods

2.1 Materials

DE was obtained from Lampang province in northern Thailand. It had particle size ($d_{4,3}$) of 73 μm, specific surface area by BET of 46.8 m^2/g and specific gravity of 2.55. Mineralogical compositions of DE consisted of quartz, montmorillonite, muscovite, kaolinite, hematite, some semi-crystalline phase and amorphous phases. Laboratory grade sodium hydroxide (NaOH) and potassium hydroxide (KOH) and industrial sodium silicate (Na_2SiO_3) with 32.39% SiO_2, 13.44 % Na_2O and 54.17% H_2O were used.

RHA with particle size ($d_{4,3}$) of 62 μm, specific surface area by BET of 48.7 m^2/g was obtained from burning of rice husk at 700 °C in oxidation atmosphere. Amorphous silica is obtained by burning rice husk at temperature lower than 700 °C [XX: P.Chindaprasirt, P. Kanchanda, A. Sathonsaowaphak and HT. Cao, Sulfate resistance of blended cements containing fly ash and rice husk ash, Construction and Building Materials, 21, 2007, 1356-1361]. XRD indicated that the mineralogical compositions of RHA composed basically of amorphous phase of silica with only small amount of cristobalite. Chemical compositions of DE and RHA are given in Table 1. Photomicrographs of DE and RHA are shown in Fig. 1.

Table 1 Chemical compositions of DE and RHA (Si/Al ratio is molar ratio of SiO_2/Al_2O_3)

Raw materials	Chemical composition (wt %)											Si/Al ratio
	SiO_2	Al_2O_3	Fe_2O_3	Na_2O	K_2O	CaO	MgO	TiO_2	P_2O_5	SO_3	LOI	
DE	77.46	10.97	9.08	tr	1.07	0.35	0.31	0.41	tr	tr	0.35	11.98
CDE800	66.48	13.18	18.31	tr	1.12	0.47	tr	0.43	tr	tr	tr	
MDE800	75.52	12.15	10.27	tr	1.24	0.39	tr	0.44	tr	tr	tr	
FDE800	79.76	10.81	7.42	tr	1.26	0.33	tr	0.42	tr	tr	tr	
RHA	85.25	0.11	0.18	tr	1.80	0.79	0.3	0.03	1.10	0.08	10.29	1315
FRHA	96.47	0.00	0.32	tr	1.50	0.75	tr	0.02	0.61	tr	tr	

Tr = trace (0-0.049%)

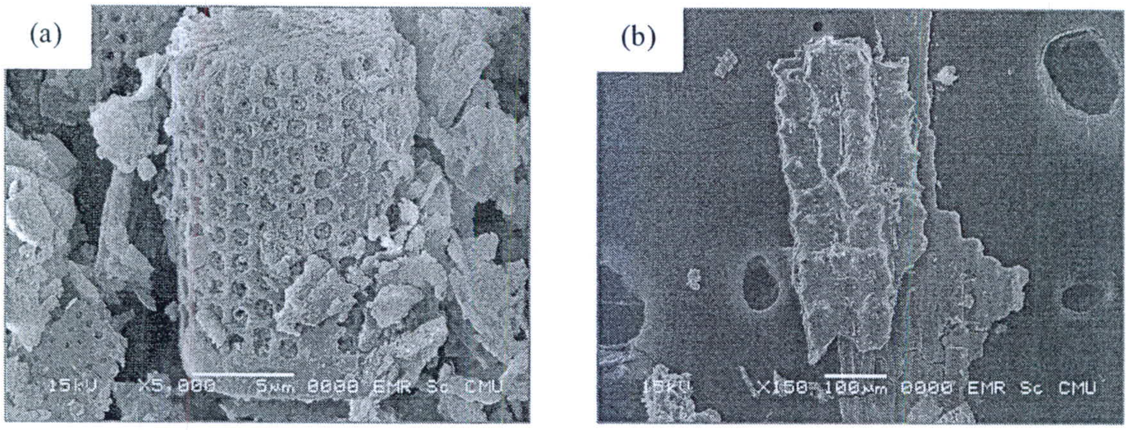


Fig.1 Photomicrographs of (a) DE and (b) RHA

DE was calcined at 400, 600, 800 and 1000 °C (DE400, DE600, DE800 and DE1000). After calcination, DE was sieved into three lots of coarse, medium and fine particle sizes. Coarse DE (CDE) was the portion larger than sieve No. 80. Medium DE (MDE) was the portion larger than sieve No. 200. Fine DE (FDE) was the portion passed sieve No. 325. Fine portion of RHA was obtained from sieving passed also sieve No. 325. The chemical compositions of DE, sieved DE800 and RHA are shown in Table 1.

The leachings of Si and Al ions of calcined DE and RHA were measured. The leaching test was carried out by stirring the calcined DE in 10 Molar (M) NaOH solution with solid to liquid ratio of 1:10 for 24 h. The dissolved Si and Al ions were verified quantitatively using Atomic Absorption Spectroscopy (AAS).

2.2 Details of tests

The mixing was done in an air conditioned room at approximately 25 °C. The proportion of each mix was stoichiometrically weighed according to Table 2. The mixing procedure started with mixing of NaOH (or KOH) solution, sodium silicate (Na_2SiO_3) and DE for 5 min. Water was then added and mixed for 5 min. The mixtures were cast in 50×50×50 mm acrylic cube mould. The specimens were then wrapped with plastic film to prevent moisture lost. After delayed time of 1 hour, the specimens were cured in an electric oven. The specimens were cooled down in the oven and then demoulded the next day and kept at 23 °C until the testing ages. The compressive strength tests were performed at 7 days in accordance with ASTM C109 [5]. Density measurements were also conducted at age of 7 days using direct measurement. The reported strength and density were the mean of three tests. XRD and SEM analysis were conducted on the broken specimens after compressive strength test using a Philips X'Celerator diffractometer with a Cu Kα_1 radiation and JSM-5910 LV microscope equipped with EDX microanalysis, respectively.

2.3 Test series

2.3.1 Effect of pre-treatment

In this series, fineness and calcination temperature of DE were studied. NaOH with concentration of 10 M and the Na_2SiO_3/NaOH ratio of 1.23 were used. After delayed time of 1 hour, specimens were cured at 60 °C for a period of 3 days.

2.3.2 Effect of synthesized condition on DE Geopolymer

The results of section 2.3.1 indicated that fine DE with optimum calcination temperature of 800 °C (FDE800) was suitable source material. It was, therefore, selected for the following tests.

(1) The concentrations of NaOH and KOH

The concentrations of NaOH and KOH of 4, 6, 8, 10 and 12 M and Na_2SiO_3/NaOH ratio of 1.23 were used. Compositions of geopolymer in mole ratios of samples No. 1 to 10 are shown in Table 2. After delayed time of 1 hour, specimens were cured at 60 oC for a period of 3 days.

(2) Curing temperature and time

The 10 M NaOH and Na_2SiO_3/NaOH ratio of 1.23 were used. After one hour delayed time, specimens were cured in an oven for different time intervals of 1, 2, 3, 4 and 5 days at curing temperatures of 60, 75 and 90 °C.

(3) Alkali content

In this test, 10 M NaOH; three Na_2O/Al_2O_3 ratios of 1, 2 and 3; and curing temperature of 60 °C for 3 days were used. Compositions of geopolymer in mole ratios of samples No. 11 to 13 are shown in Table 2.

2.3.3 Effect of incorporation of RHA

The geopolymer pastes with different DE: RHA ratios were used to obtain various SiO_2/Al_2O_3 ratios. The compositions of samples No. 14 to 17 are given in Table 2.

Table 2 Compositions of FDE800 geopolymer

Sample No.	Hydroxide	Molar	Mole ratio			
			SiO_2/Al_2O_3	Na_2O/Al_2O_3	Na_2O/SiO_2	H_2O/Na_2O
1	NaOH	4	13.0	0.68	0.05	19.53
2		6	13.0	0.79	0.06	16.39
3		8	13.0	0.90	0.07	13.94
4		10	13.0	1.00	0.08	12.16
5		12	13.0	1.09	0.08	10.86
6	KOH	4	13.0	0.66[a]	0.05[b]	19.53[c]
7		6	13.0	0.76	0.06	16.20

8		8	13.0	0.84	0.06	13.96
9		10	13.0	0.94	0.07	11.89
10		12	13.0	1.02	0.08	10.53
11	NaOH	10	13.0	1.00	0.08	12.16
12		10	13.0	1.50	X	Y
13		10	13.0	2.00	0.15	11.57
14		10	13.0	3.00	0.23	11.38
15	NaOH	10	16.6	1.25	0.08	12.16
16		10	22.5	1.67	0.07	12.16
17		10	33.5	2.46	0.07	12.04

3. Results and Discussion

3.1 Dissolubility of DE and RHA

Results of dissolubility of DE and RHA in 10M NaOH activation are shown in Table 3. The dissolution was related to the particle size and fineness of DE. The amount of leached silicon ion of CDE was 2040 mg/L and increased to 2110 and 2750 mg/L for finer DE and FDE. The finer DE possessed high surface area and allowed greater contact with the alkali solutions. Furthermore, the fine DE also contained high silica content (Table 1). For the calcined DE, the dissolution of silica and alumina were significantly affected by the calcination temperature. The dissolved silicon ions of the DE400 (DE calcined at 400 °C), DE600 and DE800 were 1690, 3540 and 4140 mg/L while those of alumina were 158, 1010, 1260 mg/L, respectively. High calcination temperature of 600 and 800 °C were need for the high dissolution of alumina ions.

The silica and alumina contained in clay minerals contributed to the increase in dissolved ions. The silica phase contained in DE was slightly changed with heat treatment. Kaolinite and montmorillonite could transform to metakaolin and disrupted montmorillonite with more active surfaces. Dissolution of silicate molecules at high pH (>10) resulted in the formation of silicic acid; $H_3SiO_4^-$, $H_2SiO_4^{2-}$, $HSiO_4^{3-}$ and SiO_4^{4-}, dependent on pH of the system [X]. The measured pH of this experiment was around 14. Thus, the silicic acid species of geopolymeric sytem were normally $H_3SiO_4^{3-}$ and SiO_4^{4-}. To these respects, the DE800 was screened through 325 mesh size sieve and used to prepare geopolymeric materials for further tests.

Table 3 Contents of aluminum and silicon ions leached from DE and RHA

Materials	Silicon ion (mg/L)	Aluminum ion (mg/L)	SiO_2/Al_2O_3 molar ratio
As-received DE	2090	219	
CDE	2040	296	
MDE	2110	230	
FDE	2750	205	
DE400	1690	158	
DE600	3540	1010	
DE800	4140	1260	
RHA	4840	0	
FRHA	5310	0	

3.2 Effect of pre-treatment on DE Geopolymer

3.2.1 Fineness of DE

The compressive strength and bulk density of geopolymer pastes with the difference finenesses are shown in Fig. 2. The strength development at 7 days of R80DE, R200DE and P325DE geopolymer pastes was 2.0, 5.0 and 6.6 kg/cm^2 respectively. The strength increases with an increase in the DE fineness. The reaction of DE geopolymer includes dissolution and polymerization [6]. DE with higher surface area would undergo a faster dissolution. This would increase the amount of silicon and aluminum ions in the solution and thus increases the strength of the paste. Data of bulk density clearly testified this trend.

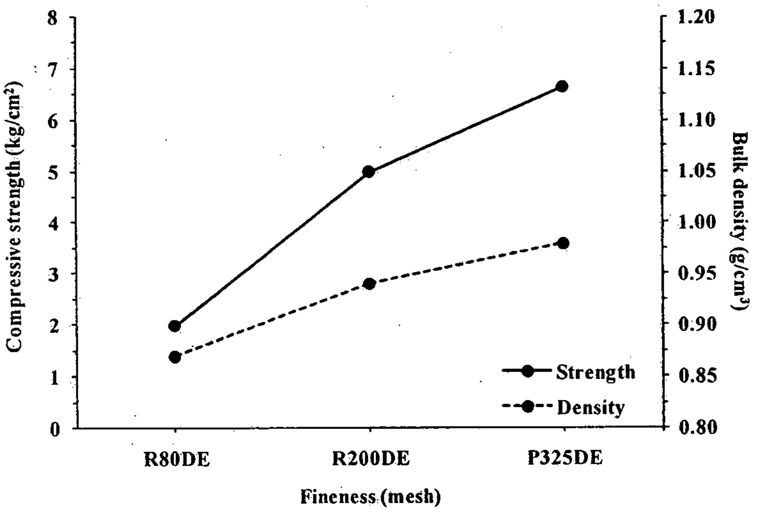
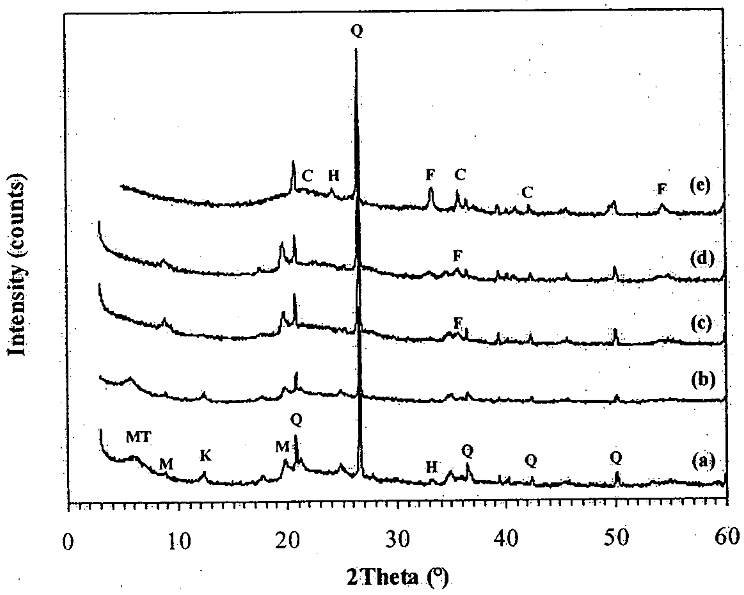


Fig. 2 Compressive strength and bulk density of geopolymer pastes with different finenesses of DE

3.1.2 Calcination temperature of DE

XRD patterns with different calcination temperatures of DE are shown in Fig. 3. The destruction of chemical structures of monmorillonite and kaolinite containing in 600, 800 and 1000 °C calcined DE are observed from the disappearances of peaks at 2θ of 6.5° and 12.5°.

Hematite (Fe_2O_3) is transformed into magnetite (Fe_3O_4). Fig. 4 shows the results of compressive strength and bulk density of geopolymer pastes made of calcined DE. The strength of DE800 paste was significantly higher than those of noncalcined DE, DE400, DE600 and DE1000 °C. At low calcined temperature, the dissolution of silicon and aluminium ions was small. At high temperature of 1000 °C, quartz was transformed to cristobalite and the magnetite phase was also increased at the reactivity of DE1000, greatly reduced. Therefore, the strength of geopolymer made of DE1000 was relative low.



MT=Montmorillonite, M=Muscovite, K=Kaolinite, Q=Quartz, H=Hematite, F=Magnetite, C=Cristobalite

Fig. 3 XRD patterns of calcined DE (a) noncalcined DE (b) DE400 (c) DE600 (d) DE800 (e) DE1000

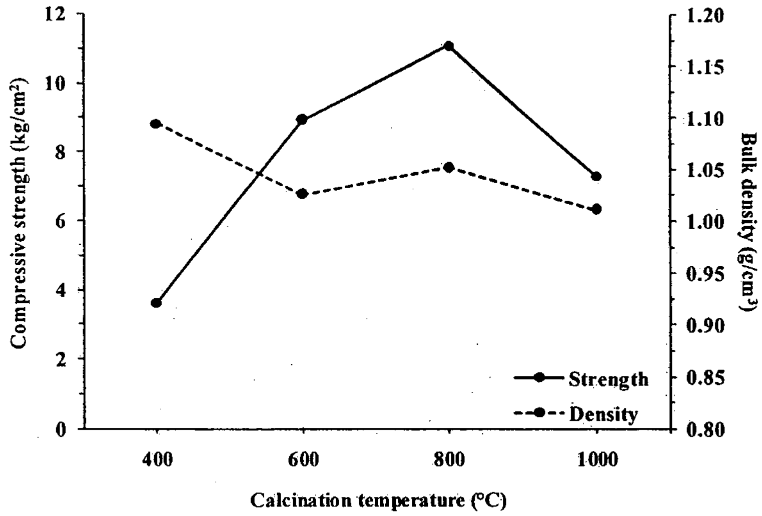


Fig. 4 Compressive strength and bulk density of geopolymer pates with different calcination temperatures of DE

3.2 Effect of synthesized condition on DE Geopolymer

3.2.1 Concentration of NaOH or KOH

Fig. 5 shows the results of compressive strength and bulk density of geopolymer with NaOH and KOH as alkaline activators. The mechanical properties increased along with an increase of concentration of NaOH or KOH. Hydroxide solution with higher concentration provided better dissolving ability of DE particles and increased production of more reactive bonds for the monomer, which increased geopolymerization of pastes [7]. When the concentration of hydroxide exceeded 10 M, the setting time was very fast. At high alkali concentration, excess hydroxide ion concentration caused aluminosilicate gel precipitation at the very early stages, resulting in lower strength geopolymer. [W.K. Lee, J.S.J Van Deventer, The effects of inorganic salt contamination on the strength and durability of geopolymer, Colloids Surf., A. 211 (2-3) (2002) 115-126]. The DE geopolymer with NaOH gave higher strength than that with KOH. Cation size of Na^+ was smaller than K^+, thus Na^+ provided higher bond strength than that of K^+.

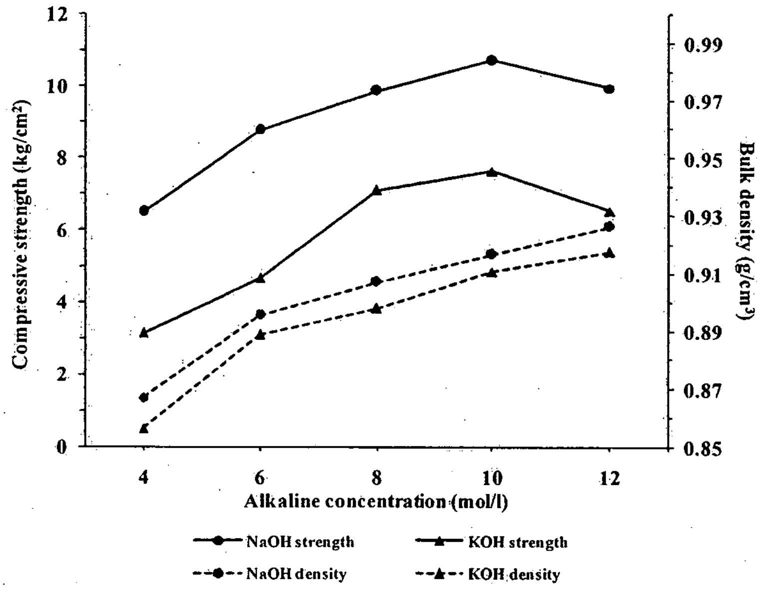


Fig. 5 Compressive strength and bulk density of geopolymer with different concentrations of NaOH or KOH

3.2.2 Curing temperature and time

The results of compressive strength and bulk density with different curing temperatures and times are given in Fig. 6. The optimum curing temperature and time were found to be 75 °C and 4-5 days. The strengths increased with the increases in curing temperature and time. For the high curing temperature of 90 °C, the strength after 5 days curing dropped. At high curing temperature, the sample could experience a substantial loss of moisture and the deterioration of strength would [8]. As expected, the density of geopolymer decreased with the increases in curing temperature and time. With the progress of polymerization, water was gradually removed and the SiO_4 and AlO_4 tetrahedral were linked to form amorphous geopolymers. The sample cured at 75 °C for 5 days has bulk density of 880 kg/m^3 while the strength was reasonably high at 1.5 MPa.

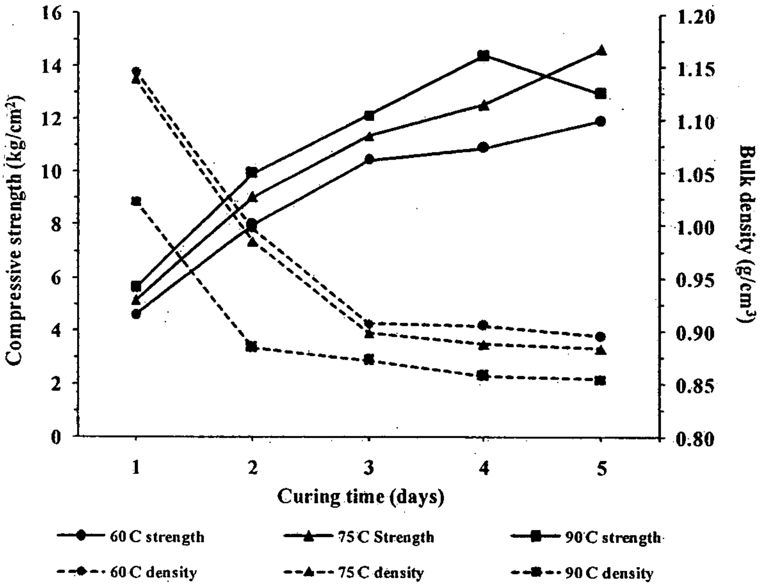


Fig. 6 Compressive strength and bulk density of geopolymers with different curing temperatures and times

3.2.3 Alkali content

Fig. 7 shows the compressive strength and bulk density of geopolymer pastes with varying Na_2O/Al_2O_3 molar ratios of 1-3. The strength increased with an increase in Na_2O/Al_2O_3 ratio. However, high alkali content (Na_2O/Al_2O_3 ratios of 2 and 3), makes polymerized networks less stable than Na_2O/Al_2O_3 ratio of 1 [XXX]. The density of geopolymer increased with an increase in Na_2O/Al_2O_3 ratio. The increase in the density of geopolymer paste to this level was undesirable.

Fig. 8 demonstrates morphological aspects of geopolymer pastes at different Na_2O/Al_2O_3 ratios. The geopolymer paste with Na_2O/Al_2O_3 of 1 was less dense than those with Na_2O/Al_2O_3 ratios of 2 and 3. Some voids and unreacted DE could be observed in the sample with Na_2O/Al_2O_3 of 1 (Fig. 8(a)). The dense amorphous glassy geopolymer matrices could be seen for Na_2O/Al_2O_3 of 2 and 3 geopolymer (Fig. 8 (b) and (c)). The results of EDX analyses of the DE geopolymers are also shown in Figs. 8 (a), (b) and (c). The major elements are Si and Al with the presence of a small amount of Na, K and Ca. Although the starting $SiO_2/Al2O_3$ ratios of DE geopolymers were similar at 13.0, the ratios of SiO_2/Al_2O_3 for DE geopolymers obtained with different Na_2O/Al_2O_3 ratios were significantly different. The Si/Al ratios for DE geopolymers with Na/Al ratios of 1, 2 and 3 were 9.9, 13.9 and 17.4 respectively. The results suggested that the dissolubility of silica and alumina were dependent on Na/Al ratios [U. Rattanasak, P. Chindaprasirt, Influence of NaOH solution on the synthesis of fly ash geopolymer, Mineral Engineering, 22, 12, 2009, 1073-1078]. It is shown here that using low Na/Al ratio (~1), the dissolubility of aluminate phases such clay minerals in DE is favorable. The higher Na/Al ratios, the silicate phases are more susceptible in dissolving. In addition, it was shown that excess sodium ions were likely more localized in the geopolymer structure with higher used Na/Al ratio. The characteristic of EDX peaks of geopolymer pastes with Na/Al ratio of 2 was very similar to that of zeolite material which is a particulate material.

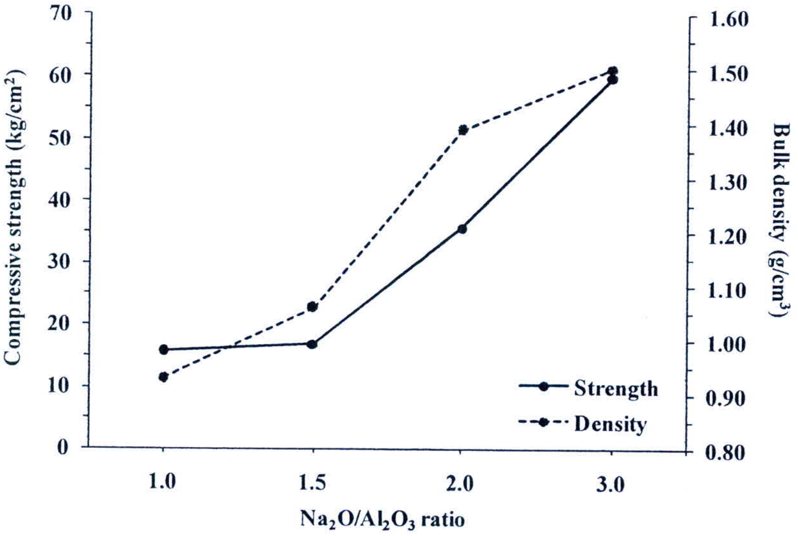


Fig. 7 Compressive strength and bulk density of geopolymer with different Na/Al ratios.

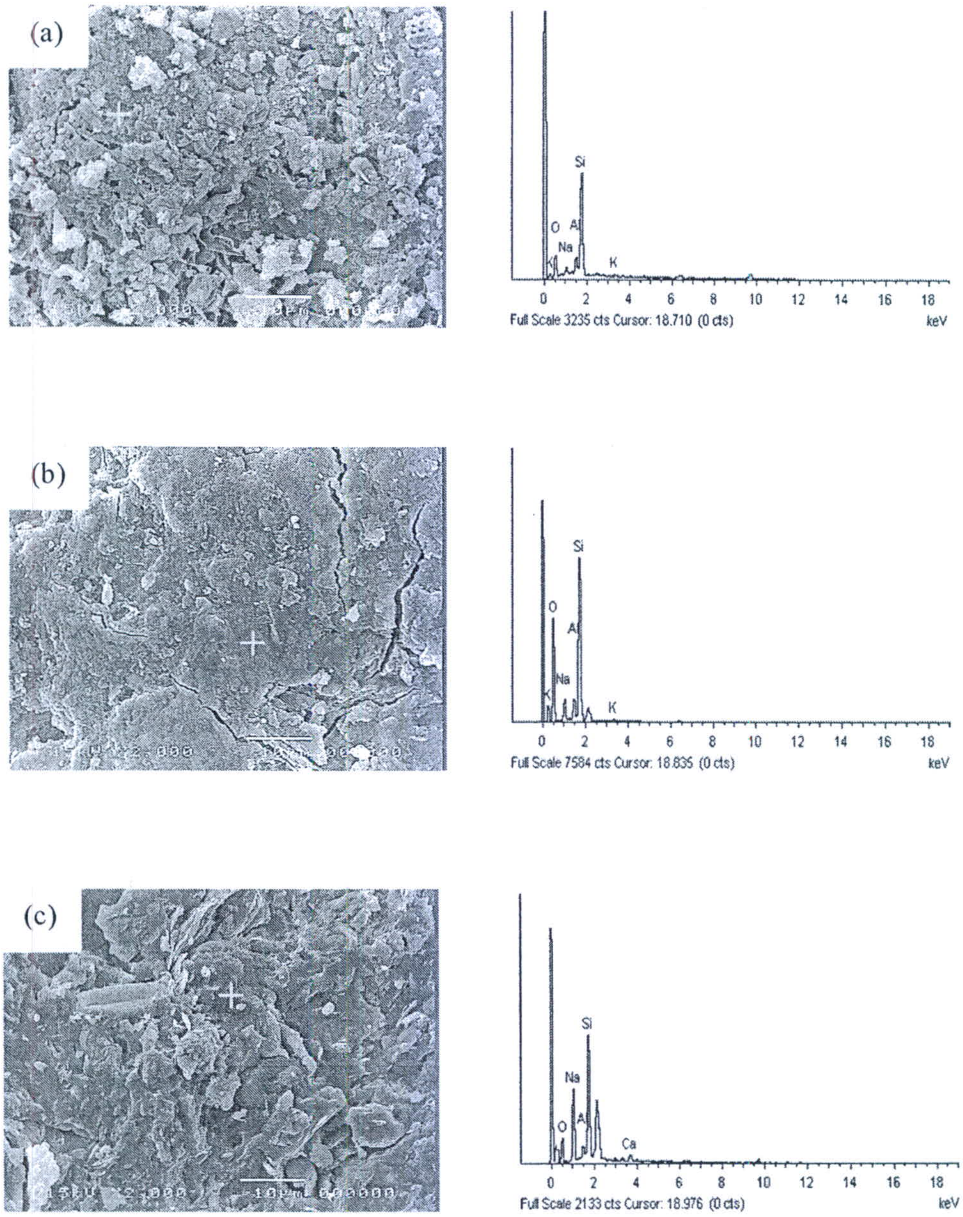


Fig. 8 SEM-EDX analyses of geopolymer pastes (a) $Na_2O/Al_2O_3 = 1$ (b) $Na_2O/Al_2O_3 = 2$ (c) $Na_2O/Al_2O_3 = 3$

Fig. 9 demonstrates the molecular bonding of geopolymer pastes at different SiO_2/Al_2O_3 ratios.

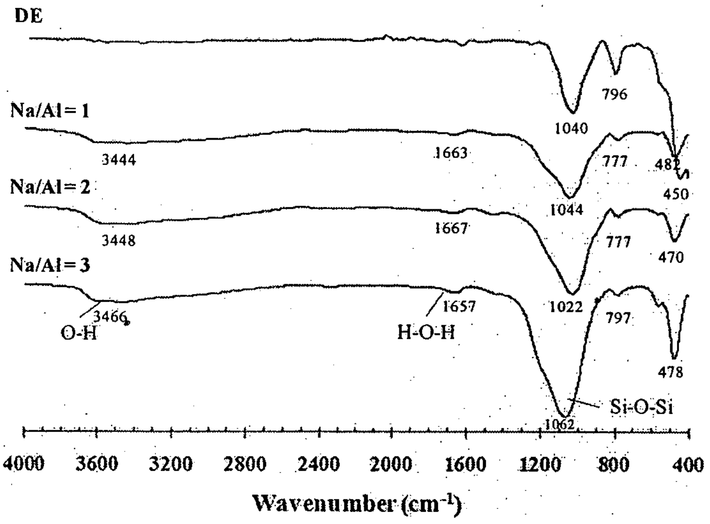
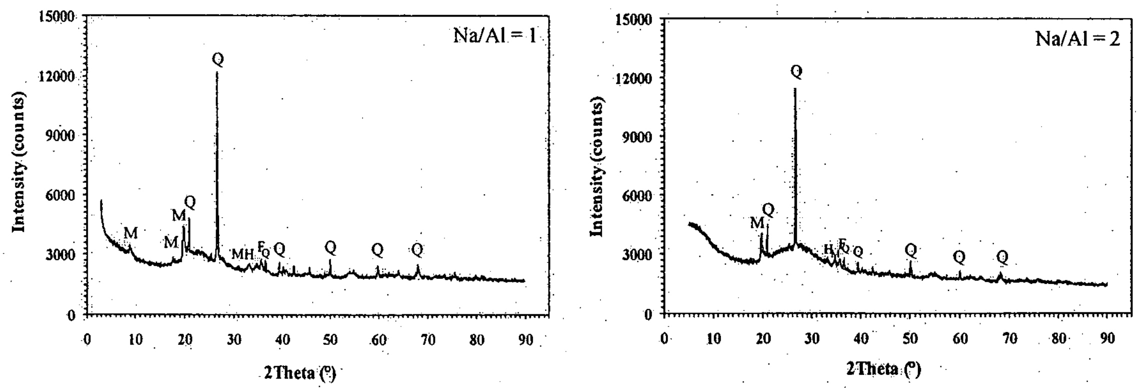
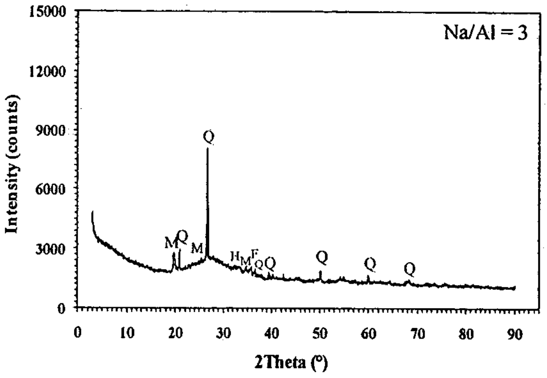


Fig. 9 IR spectra of diatomaceous earth and geopolymer with different Na/Al ratios.

Table 4 Peak area ratio from IR spectra of pastes at Si-O-Si stretching

Sample	Location of Si-O-Si (cm^{-1})	Corrected area	Peak area ratio
DE	1040	8.41	1
$Na_2O/Al_2O_3 = 1$	1044	8.70	1.03
$Na_2O/Al_2O_3 = 2$	1022	10.45	1.24
$Na_2O/Al_2O_3 = 3$	1062	15.14	1.80





M=Muscovite, Q=Quartz, F=Magnetite

Fig. 10 XRD patterns of DE geopolymer pastes with different Na/Al ratios.

3.3 Effect of proportion of DE and RHA

The RHA was used to adjust the dissolution of silica phase. The Si/Al ratios of 16.6 to 33.5 of samples No. 15 to 17 are shown in Table 2. Fig. 11 shows the compressive strength and bulk density of geopolymer pastes made of the mixtures of DE800 and RHA. The strengths increased with the increase in the amount of RHA up to 40% of solid powder which corresponded to Si/Al ratio of 22.5. The strength increased to 2.4 MPa due to the proper obtained –Si-O-Al- bonds in geopolymeric structures. An increase in the replacement level of RHA beyond this level resulted in a decrease in the strength. This is due to the obtained -Si-O-Al- bonds of high Si/Al ratio which did not favor for a condensation as long chain polymeric structure [XXXX]. Thus, they acted as defect sites and exerted a negative effect on strength of geopolymeric materials. Although, the strength increased, the density values were low at 925-1010 kg/m^3 which were highly desirable.

Fig. 12 shows XRD patterns of geopolymer pastes with 0, 20, 40 and 60% RHA. The degree of crystalline structure decreased with the replacement of DE with RHA. This could be seen at the hump peaks around 18-30 degree increased with the increase in the RHA content as a result of increases in the glassy phase. The geopolymer pastes contained similar crystalline phases to those of original DE and RHA. Quartz, muscovite, magnetite and cristobalite which were originally found in DE and RHA were undissolvable. No new crystalline phase was observed, but the increase in RHA content. The broad below 8o of 2θ was evident with the increase in RHA content. This peak suggested the formation of a mesoporous material of poorly crystalline nature. [E. Alvarez-Ayuso, X. Querol]. The increase in the glassy phase of geopolymer pastes was responsible for the increase in strength.

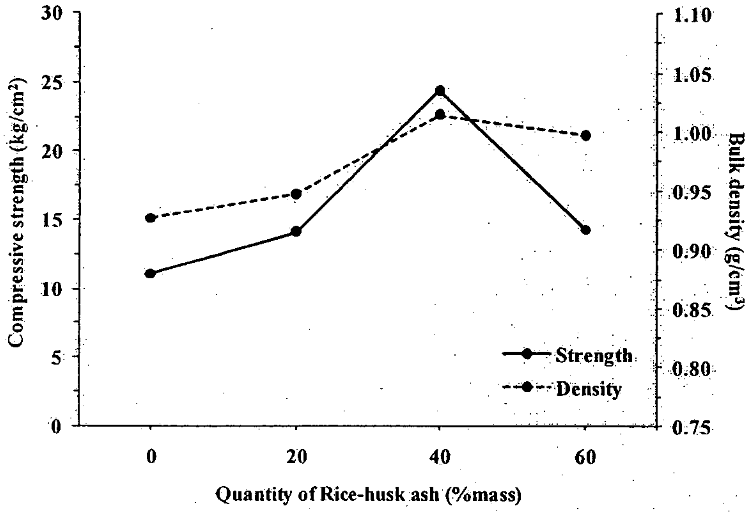
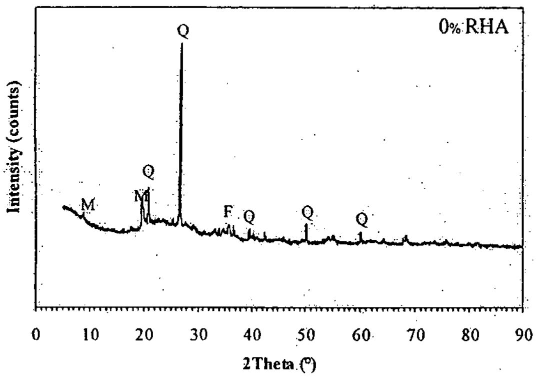
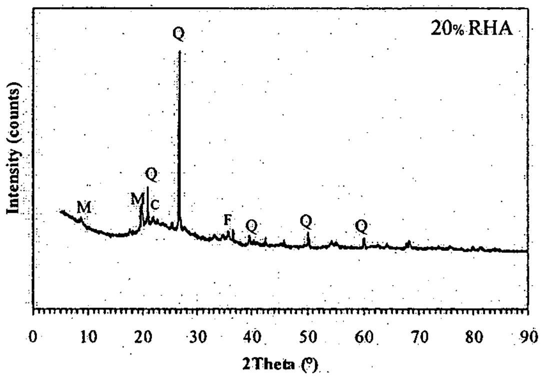
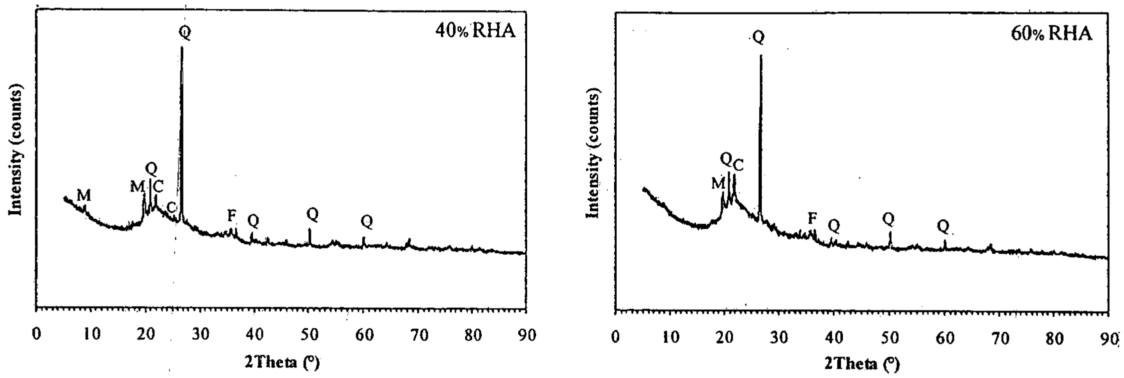


Fig. 11 Compressive strength and bulk density of geopolymer pastes with different RHA contents.







M=Muscovite, Q=Quartz, F=Magnetite, C=Cristobalite

Fig. 12 XRD pattern of geopolymer pastes with 0, 20, 40 and 60% RHA

4. Conclusions

Based on the results of investigation, the following conclusions could be drawn. DE could be used to produce lightweight geopolymeric material. The DE should be calcined and sieved to obtain fine reactive particles suitable for use as a source material. The fine DE was more reactive due to the increase in the surface area. This allowed greater contact of DE particle surface with alkali solution and hence a faster leaching of silica and alumina. The XRD patterns of the clacined DE indicated the transformation of chemical structures of montmorillonite and kaolinite. The optimum calcinations temperature of diatomaceous warth was 800 °C. With regards to the types of alkali, geopolymer pastes activated with 10M NaOH possessed higher compressive strength than that with 10M NaOH. Curing temperature and duration also affected the properties of the geopolymers. The optimum curing temperature and time were 75 °C and 5 days. The Na_2O/Al_2O_3 ratios of the mixtures also affected the properties of the geopolymer pastes. At SiO_2/Al_2O_3 ratio of 13.0, the increase in Na_2O/Al_2O_3 ratios from 1.0 to 3.0 increased the compressive strength from 1.1 to 6.0 MPa. However, the bulk density values also increased from 0.93 to 1.5 g/cm^3. To maintain the low density of geopolymer pastes, RHA was incorporated to the mixed.

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