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Lightweight bricks made of diatomaceous earth, lime and gypsum

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Abstract

Diatomaceous earth from Lampang Province in the north of Thailand composes of diatom, kaolinite, montmorillonite and illite, and has porous cellular structure. In this work, the diatomite, hydrated lime and gypsum are the main ingredients in making autoclaved lightweight bricks. Water content, pre-curing period, lime content, gypsum content and calcined temperature are the factors investigated. Mechanical and thermal properties are used to indicate their quality. The nature of hydration products and morphological characteristics of the lightweight bricks are also investigated.

The results show that the diatomaceous earth possesses pozzolanic property and can be used for making lightweight bricks. The bricks made with untreated diatomite with 15% lime and 5% gypsum shows reasonably high strength of 14.5 MPa and low density of 0.88 g/cm^3. Higher strength (17.5 MPa) and lower density (0.73 g/cm^3) are obtained with the use of diatomite calcined at 500 °C. The incorporation of lime and gypsum with a Ca/Si ratio of 0.55, 50% water content and 6 days pre-curing results in formations of calcium silicate hydrate and gismondine which enhance strength development. Calcination of diatomaceous earth results in dehydroxylation of the clay minerals and contributes to additional pozzolanic reaction and better mechanical and thermal properties of the lightweight brick.

Keywords: Diatomaceous earth; Lightweight brick; Calcium silicate hydrates; Pozzolanic reaction; Alumino-calcium-silicate-hydrate

1. Introduction

The deposit of diatomaceous earth clay or diatomite in Lampang province in the north of Thailand is quite large with estimation at more than 100 million tons. Its beneficial characteristics are light in weight due to high porosity, relatively attractive in dark-yellow shade due to high content of ferrous compound and semi-crystalline siliceous phase similar to opal ($SiO_2 \cdot nH_2O$) [1]. The pore system is very fine and its thermal conductivity is low. Diatomaceous earth possesses pozzolanic properties similar to other pozzolanic materials such as fly ash and metakaolin [2]. Significant uses of diatomite are, therefore, in construction and insulation materials such as partial replacement of Portland cement and in calcium silicate brick production [3,4].

Calcium silicate brick is produced from the mixture of $Ca(OH)_2$ and amorphous silica, or alternatively natural quartz under hydrothermal conditions [5]. The nature of products synthesized using amorphous silica is calcium silicate hydrate (CSH) gel while the product of crystalline silica is tobermorite. The hydration products are thus dependent on reactivity of starting material. By using amorphous silica, silicon ion can be readily leached out leading to a decrease in the ratio of Ca/Si of hydration products. The type of CSH phases plays a crucial role in strength development of the final product owing to their different van der Waals forces in colloidal domains [6]. The variation of Ca/Si ratio directly affects the type of CSH [7]. The formation of CSH phases takes place in aqueous solution and therefore the solubility of individual components heavily influences the formation of new phases. At the surface of silica particles, a small amount of SiO_2 dissolves and reacts with calcium ions to generate CSH phases [8]. The strength of calcium silicate bricks developed at low temperature is still low due to formation of CSH-I and CSH-II semi-crystalline structures. Therefore, hydrothermal process is necessary for the synthesis to transform amorphous phases into tobermorite [9]. Not only CSH is formed, but various types of alkaline alumino-calcium-silicate-hydrate compounds are also developed depending on the ratio of Ca, Al, and Si ions [10].

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Lampang diatomaceous earth (LDE) contains not only silica (SiO_2) but also some clay minerals [11]. These clay minerals possess low degree of reactivity. To transform them into pozzolans and to speed up its chemical reaction, it is necessary to remove hydroxyl groups from clay structures and other organic substances from clay particles by calcining.

Several other factors also influence the properties and performance of LDE. The study on lightweight bricks made of LDE should reveal its potential uses as source material for making lightweight bricks. The main aim of this study is to obtain low density and strong brick as required by any standard of lightweight constructional material. The obtained knowledge should also be useful for other research and application work in related fields.

2. Materials and methods

2.1. Materials

Diatomaceous earth was obtained from Lampang province in the north of Thailand. The chemical compositions are shown in Table 1. Commercially available hydrated lime ($Ca(OH)_2$) and gypsum ($CaSO_4 \cdot 2H_2O$) were used for this experiment. Specific gravities (S.G.) of diatomite, hydrated lime and gypsum are 0.7, 2.27 and 2.33, respectively.

2.2. Brick sample preparation

The ingredients were mixed to form uniform mixtures and then pressed at 3.5 MPa to form 3.5 cm × 7.5 cm × 15.0 cm bricks. Pre-curing at room temperature (27–28 °C) in moist cabinet at over 90% RH was carried out to allow green bricks to gain some strength before subjecting to high pressure under steam to avoid cracking. The pre-cured bricks were then autoclaved at 0.14 MPa and 130 °C in a saturated steam environment for 4 h. Several factors viz., water content, pre-curing period, lime content, gypsum content, and calcinations temperature were studied:

(a) *Water content*. LDE and lime at the ratio of 85:15 (Ca/Si = 0.23, molar ratio) with 45, 50, 55 and 60% water content and pre-curing for 6 days were used.

(b) *Pre-curing period*. LDE and lime at the ratio of 85:15 (Ca/Si = 0.23) were used with 50% water content. Pre-curing in moist cabinet for 1, 2, 3, 5, 6, 7 and 11 days were carried out.

(c) *Lime content*. To study the influences of lime content, the amount of lime were varied between 10 and 50% by weight to obtain Ca/Si ratios of 0.14, 0.23, 0.32, 0.55, 0.86 and 1.30. Water content of 50% and pre-curing of 6 days were used.

(d) *Gypsum content*. The influence of gypsum addition on microstructures of pozzolanic reaction products was also studied. Gypsum was added to the mixtures at LDE:lime:gypsum ratios of 80:20:0 (Ca/Si = 0.32), 80:15:5 (Ca/Si = 0.32), 75:15:10 (Ca/Si = 0.42) and 70:15:15 (Ca/Si = 0.55). Water content of 50% and pre-curing of 6 days were used.

(e) *Calcinations*. In addition, calcined LDE (CLDE) using a batch furnace at temperatures between 200 and 700 °C for 4 h were used to investigate the effect of calcination on pozzolanic reaction. In this test, CLDE:lime:gypsum ratio of 80:15:5 (Ca/Si = 0.32) was used as its bulk density is low. Water content of 50% and pre-curing of 6 days were used.

Table 1
Oxide compositions of LDE

SiO_2	77.5
Al_2O_3	14.16
Fe_2O_3	5.24
K_2O	2.08
Na_2O	0.20
CaO	0.00
MgO	0.46
TiO_2	0.53
P_2O_5	0.25
SO_3	0.01

2.3. Testing methods

(a) The plastic limit (PL), liquid limit (LL), and plasticity index (PI) were obtained using Atterberg limit test as described by ASTM D 4318 [12]. Plastic limit is the boundary between plastic and semi-solid states. Dry clay powder was mixed with water to produce a coherent 3 mm diameter plastic clay thread. The PL is the water content of the plastic clay when water is not sufficient to form this thread. The liquid limit is the boundary between the liquid and plastic states obtained using a Casagrande device. The plasticity index is calculated as PI = LL − PL.

(b) Electrical conductivity (EC) and pH level of LDE were determined. The conductivity is a fundamental property of a material and is defined as the conductance in Siemens per length of sample. The sample was prepared using the ratio of solid to water as 1:10 by weight. The suspension was stirred vigorously by magnetic stirrer for 10 min and then measured the EC and pH using the conductivity tester and pH tester, respectively.

(c) Specific gravities, specific surface area, chemical composition and mineralogical composition of as-received LDE were determined using pycnometer, Brunauer–Emmett–Teller (BET) method, X-ray fluorescence (XRF) and X-ray diffraction (XRD), respectively.

(d) Differential thermal analysis (DTA) and thermal gravitation analysis (TGA) were performed on LDE to investigate its physical and chemical changes during heating.

(e) Mechanical properties such as compressive strength, water absorption and bulk density of the autoclaved bricks were measured in accordance with ASTM C 170 [13] and C 373 [14], respectively. After the strength test, the tested samples were used for the studies of mineralogical and morphological compositions using XRD and scanning electron microscope (SEM).

(f) Shrinkage was determined by direct measurement of a sample length before and after autoclaving.

(g) Thermal conductivity of lightweight brick was measured using heat conduction unit (WL 370, GUNT, Hamburg, Germany). The brick samples were cut into the disc shape with diameter of 2.4 cm and 0.4 cm thick. The applied power was 4 W for the measurement.

3. Results and discussions

3.1. Properties of as-received and calcined LDE

The LDE contains cylindrical-shaped particles with square cellular structure and its fragments with d_{50} of 32 μm as shown in Fig. 1. Major mineral phases are alpha quartz, kaolinite, montmorillonite and illite. EC and pH of the diatomite are 5170 μS/cm and 7.8, respectively. This EC value is similar to that of a pozzolan such as a fly ash reported by Mccarter and Tran [15] and much higher than the EC of locally available clays deposited in Lampang basin [16]. It represents that LDE contains water-soluble compounds incorporating with some soluble salts commonly located in clay minerals. The major oxides, as shown in Table 1, are silica, alumina and ferric oxide. The silica and alumina contents are 77.5 and 14.6%, respectively and are in accordance with the requirement of natural pozzolan type N per ASTM C518.

PL and LL of LDE are 50 and 56, respectively. For mixes with 85% LDE and 15% hydrated lime (Ca/Si = 0.23), PL and LL are 55 and 67, respectively. The PI of as-received LDE is 6 while that of 85% LDE and 15% hydrated lime is 12. An increase of lime content produces an increase in PI for quartzite system. This is due to the silica–water interaction that acquires the negative charged surfaces [17]. The diatomite, by virtue of its very high surface area (BET: ~17,000 m^2/kg) comparable to microsilica [15] therefore can strongly fix the Ca^{2+} contributing electrical double layers. As Ca ion can generate the narrow double layer thickness, the strong interaction of diatomite particles takes place and thus plasticity index enhance. The increase of PI helps in resisting crack forming after pressing due to a decrease in residual stress.

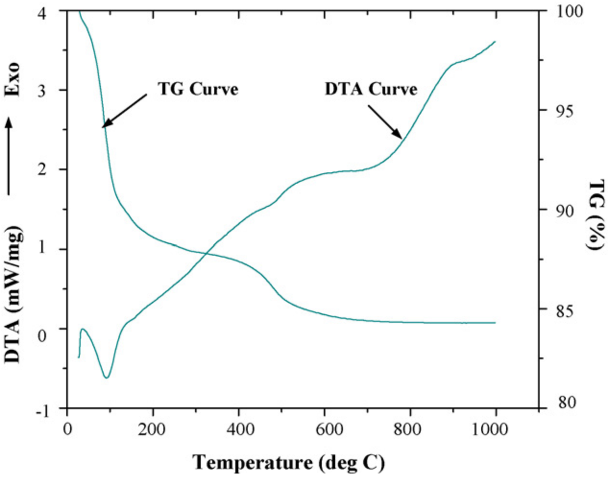


Fig. 2. DTA and TG curves of LDE.

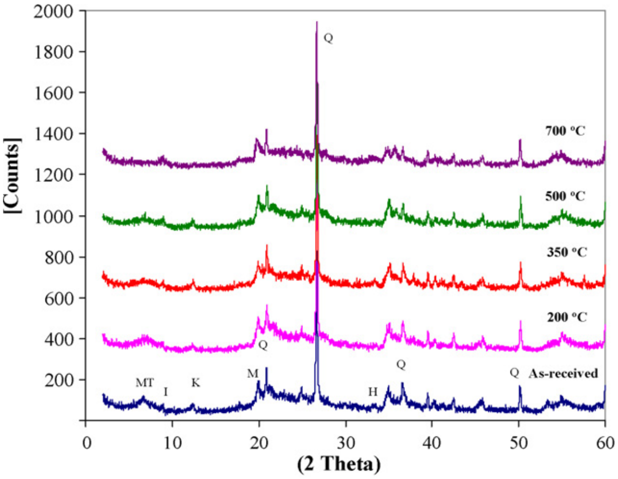


Fig. 3. XRD patterns of LDE with different calcined temperatures. H = heamatite; I = illite; K = kaolinite; M = muscovite; MT = montmorillonite; Q = quartz.

Fig. 2 shows the results of DTA and TGA of LDE on heating. Endothermic peaks are observed at 92 °C and 490 °C with corresponding mass losses of 10 and 3.5%, respectively. The peaks are ascribed to losses of adsorbed water on particle surfaces of LDE and dehydroxylation of clay minerals containing in LDE, respectively. Phase transformations on calcinations monitored by XRD of cooled products are shown in Fig. 3. At 700 °C, dehydroxylation of kaolinite and montmorillonite results in the collapses of clay mineral structures and left with illite and muscovite as confirmed by the disappearances of montmorillonite and kaolinite main peaks at 6.5° and 12.5° (2θ), respectively. Calcined clay

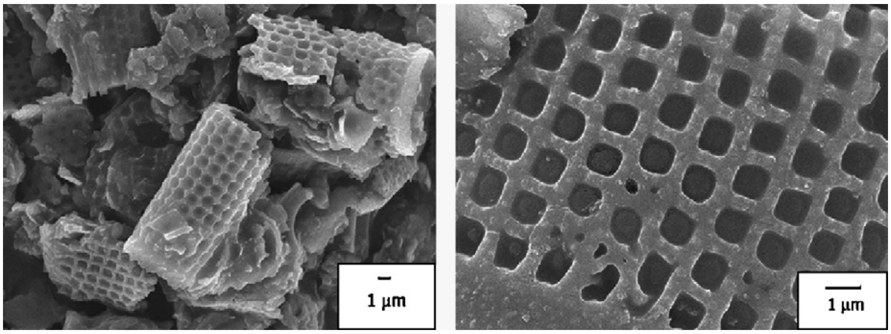


Fig. 1. SEM photomicrographs of as-received LDE particles.

Table 2
Mechanical properties of bricks with different water content

Water content (%)	Dry density (g/cm^3)	Water absorption (%)	Shrinkage (%)	Compressive strength (MPa)
45	1.10	62	0.48	13.0
50	1.15	63	0.48	14.5
55	0.92	68	0.50	13.5
60	0.94	72	0.56	10.0

Note: Ca/Si = 0.23 and 6 days pre-curing.

Table 3
Mechanical properties of bricks with different periods of pre-curing

Pre-curing (days)	Bulk density (g/cm^3)	Water absorption (%)	Shrinkage (%)	Compressive strength (MPa)
3	0.96	64	0.48	12.5
5	0.91	65	0.51	13.0
6	1.02	64	0.61	14.5
7	0.94	63	0.55	14.0
11	0.87	67	0.60	9.0

Note: Ca/Si = 0.23 and water content of 50%.

minerals contain highly charged surfaces, thus can be active in fixing Ca ions [18].

3.2. *Effect of water content*

The results of mechanical properties of brick with different water contents are shown in Table 2. Bricks with 50% mixing water show the highest strength of 14.5 MPa. In this case, Ca^{2+} ions are fixed to the faces of clay particles resulting in a reduction of the repulsive forces and clay particles become flocculated. Moreover, more space is sufficient for reaction products to grow undisturbed. An increase in the mixing water content beyond optimum value disperses the solid particles and increases the shrinkage due to overloading pore water [10] and thus causes the reverse compaction effect due to the widening of the electrical double layers [19]. Water/solid ratio is an important parameter for compaction and for hydration of cementitious materials.

3.3. *Effect of pre-curing period*

The results of test on mechanical properties of brick with different pre-curing periods are shown in Table 3. Pre-curing stage is needed to let green bricks obtain a certain green strength to avoid cracking when subjected to hydrothermal treatment. The strength developed by curing at ordinary temperature results from CSH gel formation. The highest mechanical strength and bulk density of brick are obtained with 6 days pre-curing. Less than 3 days pre-curing promotes the cracking within bricks during the hydrothermal process due to insufficient green strength. For longer pre-curing period, the reaction is quite advanced with more well-developed products and less water in the pore system. The CSH gel can be transformed into the new CSH phases with higher degree of crystallinity at high temperature and a sufficient amount of the pore water is required for the reaction. The length of pre-curing periods thus produces brick with lower strength.

Table 4
Properties of bricks with various Ca/Si ratios

Ca/Si	Bulk density (g/cm^3)	Water absorption (%)	Compressive strength (MPa)
0.14	1.05	67	13.0
0.23	1.02	64	14.5
0.32	1.27	47	15.5
0.55	1.29	45	16.5
0.86	1.29	41	17.0
1.30	1.38	43	17.0

Note: 50% mixing water and 6 days pre-curing.

3.4. *Effect of lime content*

The results of mechanical properties of brick with various lime contents and corresponding Ca/Si ratio are shown in Table 4. Strength and bulk density of bricks increase and water absorption decreases with an increase in Ca/Si ratio as a result

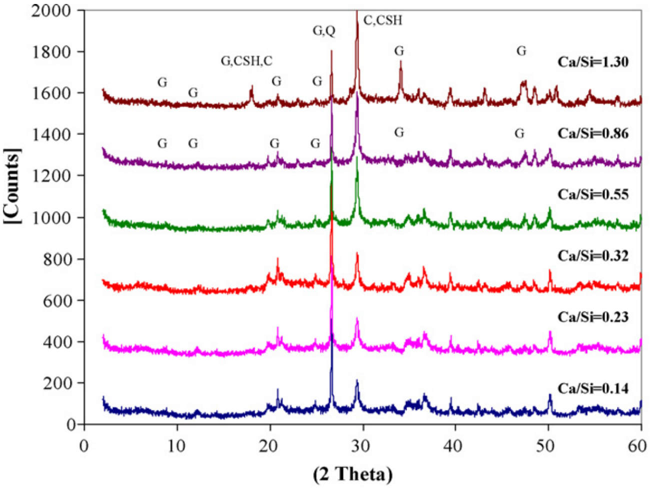


Fig. 4. XRD patterns of LDE bricks with various ratios of Ca/Si. C = calcite; CSH = calcium silicate hydrate; G = gismondine; Q = Quartz.

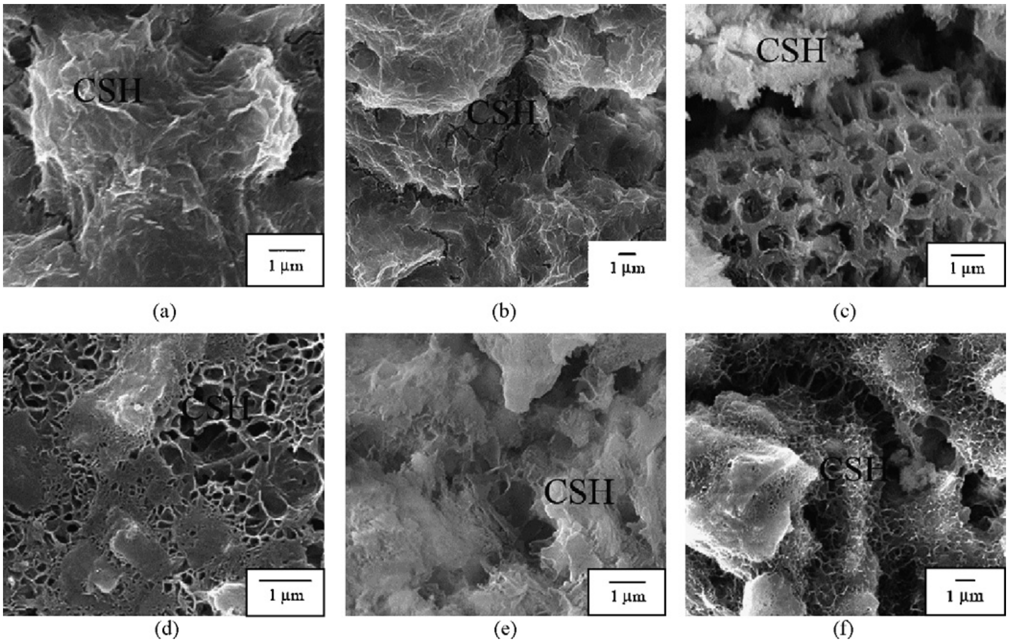


Fig. 5. SEM photomicrographs of LDE bricks with various ratios of Ca/Si: (a) 0.14; (b) 0.23; (c) 0.32; (d) 0.55; (e) 0.86; (f) 1.30.

of reductions in LDE content. Increase in strength is, however, from increases in lime content and hence additional hydration. At low Ca/Si ratio (<0.7), amorphous Z-phase CSH is obtained while at high Ca/Si ratio (0.7–1.5), 14 Å tobermorite is obtained [20] and its degree of crystallinity tends to decrease [21]. Different types of CSH are responsible for different strength behaviors due to their different characteristics. By the smaller size, the higher aspect ratio and the larger specific surface area of the particles, the higher attractive forces between particles can be obtained.

Fig. 4 shows XRD patterns of CSH compounds with Ca/Si ratio of 0.14 and greater. At Ca/Si ratio of 1.30, CSH phase is detected as a sharp peak indicating tobermorite mineral. Calcite ($CaCO_3$) is also obtained at Ca/Si ratio of 1.3 due to a carbonation of excess $Ca(OH)_2$. The strong bricks with less than 40% lime (Ca/Si < 1.30) are due to the formation of not well-developed crystalline CSH confirmed by XRD. Zeolite-like mineral named “gismondine ($Ca_4Al_8Si_8O_{32}\cdot 16H_2O$)” is also detected with Ca/Si ratio of 1.3. Normally, its production requires Ca/Si ratio of 0.5 and Al/Si ratio of 1 while it is produced with Ca/Si ratio of 0.86 in the present work. This could be due to some inert silica phases such as crystobalite contained in LDE. The dissolutions of primary quartz in LDE and clay minerals result in supersaturation of new phases as the peaks of clay minerals disappears and the intensity of quartz reduces providing the larger amount of CSH when Ca/Si ratio increases from 0.14 to 1.3.

When lime is added to clay minerals, the Ca^{2+} ions are adsorbed on clay minerals and are not available for pozzolanic reactions until clay charges are balanced with Ca^{2+} ions [22]. Therefore, there exists two chemical reactions viz., lime and silica reaction and lime and alumino-silicate reaction. As such, there are cation exchanges on montmorillonite and kaolinite surfaces as well as pozzolanic reaction between silica and alumina at their lattices. It suggests that the illite and muscovite are less involved in the pozzolanic reaction than kaolinite and montmorillonite because illite and muscovite require a higher lime content to initiate the chemical reaction. This is in a good agreement with XRD results since their peaks have collapsed when the Ca/Si ratio reaches 1.3.

The morphological characteristics of bricks are shown in Fig. 5. Fig. 5(a)–(c) show the photos of CSHs and Fig. 5(d) shows amorphous sieve-liked CSH phase. At high Ca/Si ratio of 0.86, the CSH is different from that obtained from the lower Ca/Si ratio as shown in Fig. 5(e). At very high Ca/Si ratio of 1.30, tobermorite mostly appears as shown in Fig. 5(f) similar to the findings of Zheng and Wang [23]. The Ca/Si ratio and the reactivity of primary components are important factors determining mineral composition of the final products.

3.5. Effect of gypsum

The results of mechanical properties of brick with various percentages of gypsum are shown in Table 5. For 80% LDE brick, the replacement of 5% lime with 5% gypsum results in a large reduction in bulk density and a large increase in the water absorption. Additional increase in gypsum in place of LDE results in an increase in bulk density and strength and a

Table 5
Properties of brick with different gypsum content

LDE:lime:gypsum (Ca/Si)	Bulk density (g/cm^3)	Water absorption (%)	Compressive strength (MPa)
80:20:0 (0.32)	1.27	47	15.5
80:15:5 (0.32)	0.88	66	14.5
75:15:10 (0.42)	0.97	57	15.5
70:15:15 (0.55)	1.02	32	18.0

Note: 50% mixing water and 6 days pre-curing.

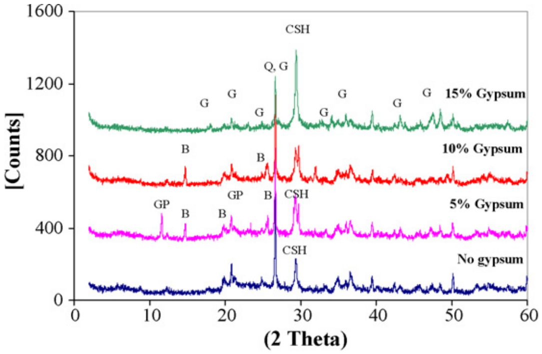


Fig. 6. XRD patterns of LDE bricks with various amount of gypsum. B = bassanite; CSH = calcium silicate hydrate; C = calcite; G = gismondine; GP = gypsum; Q = quartz.

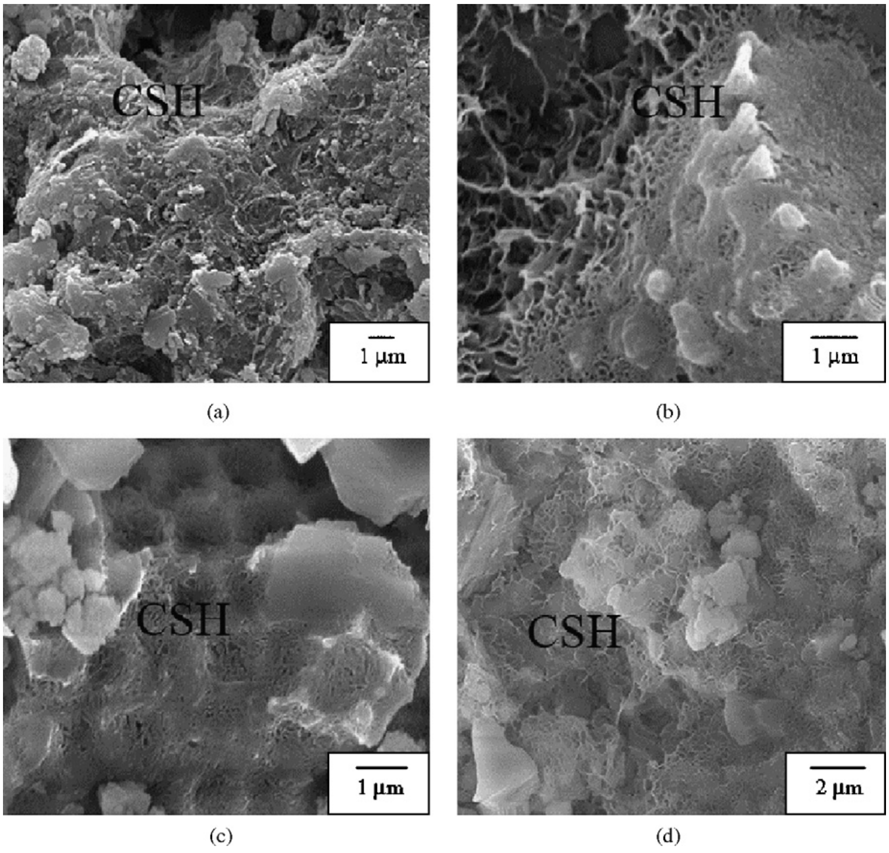
reduction in water absorption. This is because sulfate from gypsum easily attacks Al ions and produces additional hydration product that contributing to the strength development.

It has been shown that the reactions produce CSH and a small amount of gismondine at low Ca/Si ratio of 0.32. With an increase of Ca/Si ratio, the amounts of gismondine and CSH increase as confirmed by the enhancement of the intensities of XRD peaks shown in Fig. 6. At Ca/Si ratio of 0.32 with 5% gypsum addition, gypsum and bassanite are detected. It is believed that the hydrothermal temperature can transform the added gypsum into bassanite ($CaSO_4\cdot(1/2)H_2O$). At Ca/Si ratio of 0.42, only bassanite remains. At Ca/Si ratio of 0.55, both gypsum and bassanite are not detected. With an increase in gypsum content, sulfate ions can be located in CSH structure as substitution in silicate tetrahedral site. The products contain mainly CSH and gismondine resulting in relatively high strength brick. It has been reported that a low Ca/Si ratio promotes the formation of poorly crystallized CSH and prevents tobermorite formation [24]. It should be pointed out here that the left behind bassanite should not exist in a large quantity since it can hydrate and form gypsum which causes large expansion and cracks.

The morphology of bricks with gypsum is shown in Fig. 7. Again CSH phases are detected with gypsum contents of 0, 5, 10 and 15% corresponding to Ca/Si ratio of 0.32, 0.32, 0.42 and 0.55. At high Ca/Si ratio of 0.55, CSH phases as shown in Fig. 7(d) look similar to gypsum added brick at Ca/Si ratio of 0.55 which is slightly lower than 0.86 of brick without gypsum.

3.6. Effect of calcining temperature

The properties of bricks with different calcination temperatures of LDE are shown in Table 6. The use of LDE calcined at 500 °C produces bricks with high strength (17.4 MPa) and low density (0.73 g/cm^3). It is apparent that dehydroxylation of clay minerals at 500 °C contributes to the strength development. With calcination, clay mineral structures contain highly negative charged surfaces. At high calcination temperature



(a) (b) (c) (d)

Fig. 7. SEM photomicrographs of LDE bricks with various amount of gypsum: (a) no gypsum (Ca/Si = 0.32); (b) 5% gypsum (Ca/Si = 0.32); (c) 10% gypsum (Ca/Si = 0.42); (d) 15% gypsum (Ca/Si = 0.55).

Table 6
Properties of brick with different calcined temperature

Calcined temperature (°C)	Bulk density (g/cm^3)	Water absorption (%)	Compressive strength (MPa)	Thermal conductivity (W/(m K))
Uncalcined	0.88	66	14.5	0.165
200	0.76	56	15.0	0.144
350	0.79	50	15.5	0.152
500	0.73	46	17.5	0.130
700	0.76	49	12.5	0.150

Note: CLDE:lime:gypsum of 80:15:5 (Ca/Si = 0.32), 50% mixing water and 6 days pre-curing.

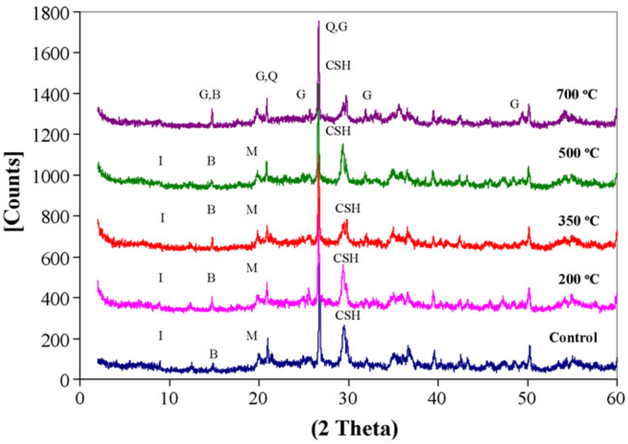


Fig. 8. XRD patterns of bricks with different calcined temperatures (Ca/Si = 0.32). B = bassanite; C = calcite; CSH = calcium silicate hydrate; G = gismondine; Q = quartz; I = illite; M = muscovite.

of 700 °C, strength reduces due to the formation of bassanite. The reactive clay minerals can be obtained between 500 and 700 °C. XRD peaks, particularly kaolinite and montmorillonite peaks, disappear in this temperature range as shown in Fig. 3. The proper calcination temperature of LDE is slightly higher than 500 °C because the most reactive phases can be attained right after the dissociation of hydroxyl ions from clay structures. The DTA and TGA curves shown in Fig. 2 indicate the proper calcination condition. With calcination, sulfate ions can be located in the structure of CSH as a silicate ion substitution while uncalcined condition has no substitution at the Ca/Si ratio of 0.32. Therefore, the calcinations is beneficial to the chemical reaction for low Ca/Si ratio. Illite and muscovite do not undergo pozzolanic reaction with lime at low Ca/Si ratio as they need high calcine temperature of 700 °C.

The reduction of bulk density with an increase in calcination temperatures is due to the increase of negatively charged clay surfaces. This results in an increase of particle–particle

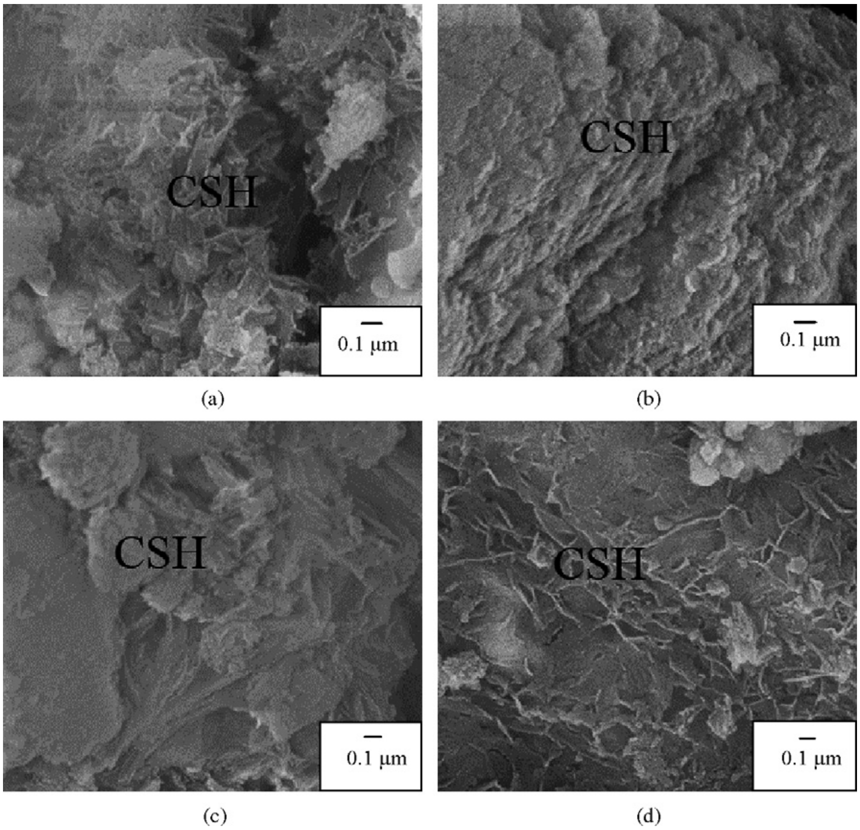


Fig. 9. SEM photomicrographs of bricks with different calcined temperatures (Ca/Si = 0.32): (a) 200 °C; (b) 350 °C; (c) 500 °C; (d) 700 °C.

repulsive forces of CLDE and the formations of pozzolanic reaction products and gismondine. An increase of bulk density is due to the reduction of the repulsive forces on account of the disappearance of meta-stable phases. The results of thermal conductivity of bricks made of CLDE are also shown in Table 6. Lightweight brick made of 500 °C CLDE gives the lowest thermal conductivity which is dependent on the density of bricks. The XRD patterns of the mixtures are dominated by remaining quartz, gismondine and CSH as shown in Fig. 8. The amount of the hydration products is optimum at calcining temperature of 500 °C and thus contributes to highest strength characteristics of brick. In addition, there is only a few bassanite left behind in the sample prepared from 500 °C CLDE because sulfate ions can substitute the silicate ions in the layer structure. The mechanical properties of the resulting bricks are very promising for building construction as standard specification of calcium silicate bricks (ASTM C 73).

The microstructures of bricks made of CLDE–lime–gypsum mixtures with different calcination temperatures are shown in Fig. 9. Microstructures of CLDE bricks are quite different from those of LDE brick. The CSH phases in LDE brick are well developed but not dense. Whereas those in CLDE bricks with gypsum are relatively dense CSH products with finer networks which is seen by relatively high resolution SEM as shown in Fig. 9.

4. Conclusion

Lampang diatomaceous earth containing some clay minerals is suitable for making lightweight bricks by autoclaving at 0.14 MPa and 130 °C in a saturated steam environment for 4 h. The mechanical properties of the lightweight brick viz., strength, water absorption, and bulk density are dependent on the amount of hydrated lime, gypsum, water content and pre-curing period. The optimum water content and pre-curing are 50% and 6 days, respectively. Increase in lime content, increase the strength and density of the brick. The use of gypsum to replace part of lime results in only a slight reduction in strength but with a significant reduction in density. The bricks made with LDE:lime:gypsum of 80:15:5 shows reasonably high strength of 14.5 MPa, with low density of 0.88 g/cm^3 and high water absorption of 66%. Better mechanical properties of brick are obtained with the use of CLDE at 500 °C for 4 h. The bricks made with CLDE:lime:gypsum of 80:15:5 shows high strength of 17.5 MPa, low density of 0.73 g/cm^3, water absorption of 46% and low thermal conductivity of 0.130 W/(m K).

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