Characteristic of calcined diatomaceous earth from Aceh Besar District – Indonesia as cementitious binder

M Hasan¹, T Saidi¹, A Muyasir¹, Y R Alkhalay² and M Muslimsyah³

¹Department of Civil Engineering, Universitas Syiah Kuala, Banda Aceh, Indonesia
²Department of Civil Engineering, Universitas Malikussaleh, Lhokseumawe, Indonesia
³Department of Architecture, Universitas Syiah Kuala, Banda Aceh, Indonesia

Corresponding e-mail: muttaqin@unsyiah.ac.id

Abstract. Diatomaceous earth is a naturally occurring, soft, siliceous sedimentary rock, which is easily crumbled into a fine powder. In this paper, the characteristic of calcined diatomaceous earth from Aceh Besar District – Indonesia as a cementitious binder is studied. The chunk of diatomaceous earth was burned in the brick burning furnace for 4 days, then ground so that it becomes a powder. The powder was sieved to form the binder having the particle sizes less than 250 μm. The binder then calcined at the temperature of 600 °C for 5 hours in a laboratory furnace. The density, specific gravity, absorption, particle size analyzer, X-ray diffraction, X-ray fluorescence, and scanning electron microscopy tests were therefore performed. The test results show that the density, saturated surface dry specific gravity, and absorption of calcined diatomaceous earth binder are 767 kg/m³, 2.000, and 6.535 %, respectively. The particle sizes range from 0.107 μm to 231 μm. The main minerals of the particle are graphite, quartz, and lead-silver thallium antimony sulphide. The main chemical compositions of calcined diatomaceous earth are SiO₂, 56.00 %; Fe₂O₃, 26.40 %; ZrO₂, 7.20 %; and Al₂O₃, 6.50 %.

1. Introduction
Many studies have lead-silver on the use of geopolymer such as fly ash, palm oil ash, rice husk ash, ground granulated blast furnace slag as cementitious materials for cement replacement in concrete production [1-7]. In recent years, the study on alkali-activated of such geopolymer materials is intensively conducted [8-12]. Diatomaceous earth, which is the silica-rich material, is one of the natural geopolymers and can be used as the supplementary cementing material. The properties of the blended cement containing diatomaceous earth have been studied [13-16]. The properties of concrete with diatomaceous earth as a partial substitution of cement have also been studied [17].

In Aceh Besar District – Indonesia, the deposit of the diatomaceous earth is quite high with an estimation of 40,353,700 tons weight. Since the main composition, the silica content and the structure of diatomaceous earth widely vary depend on its origin, the study of the characteristic of diatomaceous earth from Aceh Besar District – Indonesia as cementitious material is essential. This study aims to characterize the physical properties of calcined diatomaceous earth binder originated from Aceh Besar District – Indonesia such as density, specific gravity, absorption, particle size distribution as well as its mineralogical, crystal structure and chemical composition.
2. Methodology
The diatomaceous earth used in this study was collected from Beureunuet Village, Aceh Besar District, Indonesia, in the form of the chunk. The location where the material was collected is shown in figure 1.

Figure 1. Map of the location where the material was collected.

2.1. Preparation of material
The chunk of diatomaceous earth was burned in the brick burning furnace for 4 days. When the temperature of diatomaceous earth chunk returned to room temperature, the material was ground in the Los Angeles test device. The material was then sieved to form a binder with the size of the particle less than 250 μm. The diatomaceous binder was calcined in the laboratory furnace at the temperature of 600 °C for 5 hours for used as a cementitious material.

2.2. Density test
The density of calcined diatomaceous binder was tested according to ASTM C188-17 [18]. Three samples were prepared. The sample was filled in a container that has a volume of 1.552 liters. The weight of the sample and container was measured as well as the weight of the container only. The density of the sample was then calculated as follows:

\[
\text{Density} = \frac{(W_t - W_c)}{V_c}
\]

where \( W_t \) = weight of sample and container, \( W_c \) = weight of the container, and \( V_c \) = volume of the container.

2.3. Specific gravity and absorption tests
The specific gravity and absorption of the calcined diatomaceous binder were tested on 3 samples using a pycnometer. The specific gravity was tested on the saturated surface dry (SSD) and oven-dry (OD) conditions.
2.4. Particle size analyzer (PSA) test
Particle size analyzer (PSA) test was performed to know the particle size distribution of calcined diatomaceous binder using MicroBrook 2000 L PSA test device manufactured by Brookhaven Instruments Corporation with laser diffraction method and has measured scale range from 0.02 \( \mu \)m to 2000 \( \mu \)m. The test was repeated 3 times on 3 different samples.

2.5. X-ray diffraction (XRD) test
X-ray diffraction (XRD) test was conducted to study the crystal structure and mineralogical characterization of calcined diatomaceous earth binder. In this study, a Maxima XRD-7000 device manufactured by SHIMADZU was used. XRD device uses X-ray radiation from the XRD tube with the length of the wave of 0.154184. The resulted X-ray diffraction will be in the form of X-ray diffraction intensity and the 2\( \theta \) angles.

2.6. X-ray fluorescence (XRF) test
The chemical composition of calcined diatomaceous earth binder was characterized by X-ray fluorescence (XRF) test. An XRF Analyzer of TORONTOTECH TT-EDXPRT.XRF was used in this study for the XRF test.

2.7. Scanning electron microscopy (SEM) test
The surface texture (topography) and morphology (particle shape and size) of calcined diatomaceous earth binder were analyzed by using scanning electron microscopy (SEM) image. A JSM-6510 LA device manufactured by JEOL was used in this study.

3. Results and discussion
In this section, the characteristics of calcined diatomaceous earth obtained from the tests, as mentioned above, are described and discussed.

3.1. Density
The density of calcined diatomaceous earth binder originated from Aceh Besar District – Indonesia obtained from this study is summarized in Table 1. Table 1 shows that the calcined diatomaceous earth has a low density compared to cement (density = 3150 kg/m\(^3\)). The low density of calcined diatomaceous earth has an implication on more high volume need when it used as cement replacement in concrete production. However, if the same volume of replaced cement is used, the concrete produced with the calcined diatomaceous earth has a low density compared with normal concrete.

| Sample number | The density of calcined diatomaceous earth (kg/m\(^3\)) |
|---------------|-------------------------------------------------------|
| #1            | 760                                                   |
| #2            | 773                                                   |
| #3            | 767                                                   |
| Average       | 767                                                   |

3.2. Specific gravity and absorption
The specific gravity (SSD and OD) and absorption of calcined diatomaceous earth binder obtained from this study are summarized in table 2. Table 2 shows that calcined diatomaceous earth binder has lower specific gravity compared with Portland cement and other cementitious materials. The absorption of calcined diatomaceous earth binder is 6.535 \% which causes the more water need in mixing concrete with calcined diatomaceous earth compared to concrete with only original Portland cement.
3.3. Particle size distribution

The particle size distribution of calcined diatomaceous earth binder used in this study is shown in figure 2. The particle sizes range from 0.107 μm to 231 μm. More than 50 % of the sample has a particle diameter less than 33.90 μm. Based on the particle size distribution, the calcined diatomaceous earth binder in this study can be classified as microparticles that can be used for cement replacement in concrete production.

| Sample number | Specific gravity | Absorption (%) |
|---------------|-----------------|----------------|
|               | SSD  | OD  |                  |
| #1            | 2.000 | 1.880 | 6.383            |
| #2            | 2.000 | 1.880 | 6.383            |
| #3            | 2.000 | 1.872 | 6.838            |
| Average       | 2.000 | 1.877 | 6.535            |

Figure 2. The particle size distribution of calcined diatomaceous earth binder.

3.4. Crystal structure and mineralogical characteristics

The XRD pattern of calcined diatomaceous earth binder is shown in figure 3. The strongest 3 peaks occur at 2θ = 27.755°, 26.583°, and 29.901°. The XRD pattern shows that graphite, quartz, and lead-silver thallium antimony sulphide are the main crystalline minerals of calcined diatomaceous earth binder. In addition, an amorphous silica phase (opal A) is also present. The other minerals present in the calcined diatomaceous binder are Plustite, lead, hydroxyl, calcibet, tousonite, algodonite, lueshite, macedonite, delafossite, lenoblite, krutaita (cobalti), eul, frankdicksonite, khamrabaevite, dyscrasite, sorosite, zirconia, kurulite, tennantite, kerstenite, schreyerite, ikunolite, tetrahedrite, sein, berthie, toyohaite, sakharovai, bismutite, brunoeigerite, bunsenite, schoenfliesite, wickmanite, cuprospinel, rhenium, Plustedtite, tellurium, tellurobismuthite, mushistonite, ferruesite, arsen, ceryopiro, ferrowodginite, ferryhidrid, tsumoite, and zairite.

3.5. Chemical composition

The chemical composition of the calcined diatomaceous earth binder is shown in table 3. Based on the chemical composition, the binder can be classified as a cementitious binder with the content of SiO₂, Fe₂O₃, and Al₂O₃ is 88.90 %. According to ASTM C618-03, the binder is classified as Class N natural pozzolan [19].
Figure 3. XRD pattern of calcined diatomaceous earth binder.

Table 3. Chemical composition of calcined diatomaceous earth binder (%).

|                |       |
|----------------|-------|
| SiO₂           | 56.00 |
| Fe₂O₃          | 26.40 |
| ZrO₂           | 7.20  |
| Al₂O₃          | 6.50  |
| TiO₂           | 1.50  |
| Sb₂O₃          | 1.30  |
| MnO₂           | 0.29  |
| ZnO            | 0.27  |
| SnO₂           | 0.20  |
| NiO            | 0.11  |
| Cu₂O           | 0.05  |
| PbO₂           | 0.02  |

3.6. Scanning electron microscopy (SEM) images

The SEM images of calcined diatomaceous earth binder are shown in figure 4. The shape of calcined diatomaceous earth particles is cylindrical with a circular cellular structure. The diameter of the cellular is around 2 μm. In addition, it also contains 15 μm size cube-shaped particles.

4. Conclusion

Based on the characteristic found in this study, the calcined diatomaceous earth binder from Aceh Besar District – Indonesia can be used as a cementitious material. The characteristics are as follows:

- The density of calcined diatomaceous earth is 767 kg/m³.
- The saturated surface dry and oven-dry specific gravity is 2.000 and 1.877, respectively.
- The particle size has a diameter ranging from 0.107 μm to 231 μm. More than 50 % of the sample has a particle diameter less than 33.90 μm.
Figure 4. SEM images of calcined diatomaceous earth.

- The main minerals in calcined diatomaceous earth binder are graphite, quartz, and lead-silver thallium antimony sulphide. In addition, an amorphous silica phase (opal A) is also present.

- The main chemical compositions of calcined diatomaceous earth binder are $\text{SiO}_2$, 56.00 %; $\text{Fe}_2\text{O}_3$, 26.40 %; $\text{ZrO}_2$, 7.20 %; and $\text{Al}_2\text{O}_3$, 6.50 %.
- The shapes of calcined diatomaceous earth particles are cylindrical with 2 μm circular cellular and 15 μm size cube.

Acknowledgments
This work was supported by a grant provided by the Directorate of Research and Community Service, Ministry of Research, Technology, and Higher Education of the Republic of Indonesia under Contract No. 099/SP2H/LT/DPRM/2019 and LPPM Universitas Syiah Kuala under Contract No. 12/UN11.2/PP/SP3/2019. The authors thank Miss Nurul Husna Zubir, Miss Asmaul Husna Zubir, Miss Raudha Azzahra, Miss Putroe Nadilah Rahman, Miss Rifa Risky Ayunizar, Mr. Mahlil, Mr. M. Nasir and Mr. Razali for their assistance in experimental work.

References
[1] Sata V, Jaturapitakkul C and Kiattikomol K 2004 J. Mater. Civ. Eng. 16 623-628
[2] Chindaprasirt P, Homwutiwong S and Jaturapitakkul C 2007 Constr. Build. Mater. 21 1492-1499
[3] Haque M N and Kayali O 1998 Cem. Concr. Res. 28 1445-1452
[4] Aulia T B, Muttaqin, Afifuddin M, Zaki M, and Merriza S 2018 IOP Conf. Series: Mater. Sci. and Eng. 434 012199
[5] Hamzani, Munirwansyah, Hasan M and Sugianto S 2019 IOP Conf. Series: Mater. Sci. and Eng. 523 012034
[6] Poon C S, Lam L and Wong Y L 2000 Cem. Concr. Res. 30 447-455
[7] Hasan M, Husin S and Nursaniah C 2016 Key Engineering Materials 711 302-309
[8] Luukkonen T, Abdollahnejad Z, Yliniemi J, Kinnunen P and Illikainen M 2018 Cem. Concr. Res. 103 21-34
[9] Provis J L 2018 Cem. Concr. Res. 114 40-48
[10] Myers R J, Bernal S A and Provis J L 2017 Cem. Concr. Res. 95 30-38
[11] Ding Y, Dai J G and Shi C J 2016 Const. Build. Mater. 127 68-79
[12] Kaur K, Singh J and Kaur M 2018 Const. Build. Mater. 169 188-192
[13] Kastis D, Kakali G, Tsivilis S and Stamatakis M G 2006 Cem. Concr. Res. 36 1821-1826
[14] Stamatakis M G, Fragoulis D, Csirik G, Bedelean I and Pedersen S 2003 Cem. Concr. Compos.25 177-184
[15] Fragoulis D, Stamatakis M G, Papageorgiou D and Chaniotakis E 2005 Cem. Concr. Compos.27 205-209
[16] Papadakis V G and Tsimas S 2002 Cem. Concr. Res. 32 1525-1532
[17] Tagnit-Hamou A, Petrov N and Luke K 2003 ACI Mater. J. 100 73-78
[18] ASTM C188-17 Standard Test Method for Density of Hydraulic Cement
[19] ASTM C618-03 Standard Specification for Coal Fly Ash and Raw or Calcined Natural Pozzolan for Use in Concrete