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Properties and microstructure of composite cement paste with diatomaceous earth powder (DEP) from Aceh Besar district – Indonesia

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Abstract

The research was conducted to determine the properties of composite cement paste prepared using diatomaceous earth powder (DEP) from Aceh Besar District, Sumatra Island, Indonesia. The focus of this study was on characteristics such as water demand, setting time, flow, compressive strength, and microstructure. The process involved calcining the diatomaceous earth at a temperature of 600 ºC for 4 h, ground, and sieved with #200 to produce a powdered form. The DEP was blended with Ordinary Portland Cement (OPC) to produce a composite cement with DEP content of 0%, 10%, 20%, and 30% (w/w). The DEP, OPC, and water were mixed to produce a paste while the procedures for the test were in line with EN and ASTM Standards. Furthermore, the fresh paste was tested for the water required for normal consistency, setting time and flow while the compression for the 50 mm size cube specimens was determined at the ages of 28 and 120 days. The analysis of the microstructure at the age of 120 days was evaluated using the scanning electron microscopy (SEM) image. The water demand was found to have increased with the DEP content but the initial setting time and flow decreased. It was also discovered that 10% and 20% DEP in composite cement had almost the same compressive strength as OPC paste. Moreover, the SEM image showed the presence of $Ca(OH)_2$ in OPC paste while a massive structure of calcium silicate hydrate (CSH) was observed in the composite cement pastes with 10% and 20% DEP.

Keywords: Diatomaceous earth, Composite cement, Compressive strength, Microstructure, Water demand

1. Introduction

There are several rich mineral resources in Indonesia such as the diatomaceous earth and the information obtained from the Mining and Energy Office in Aceh province showed it has a significant deposit of approximately 40,353,700 tons in Aceh Besar District. It was also reported to contain approximately 55-70% amorphous silica as its major chemical constituent based on the area it was found. This means the silica compounds content and structure of the mineral vary widely and strongly determined by the origin [1]. Moreover, silica-rich materials can be used as supplementary cementing materials (SCM). Therefore, it was important to study the characteristics, properties, and microstructure of the diatomaceous earth from Aceh Besar District to determine its potential in producing composite cement.

Some researchers have studied the production of composite cement using the diatomaceous earth materials [2-5] and the water it required was also found to be higher in comparison with the Ordinary Portland Cement (OPC) [2-4]. Moreover, a reduction of 24.14% was recorded for the compressive strength after 28 days due to the use of approximately 35% calcareous diatomaceous rock obtained from the Ionian Sea of Zakinthos Island which majorly contains $CaCO_3$ and amorphous silica retrieved as opal-A from the biogenic origin. However, it has also been reported that the blended cement with 10% diatomite rocks developed a similar trend as observed in the OPC [2] and this means diatomite rocks have the potential to have a product with a high strength depending on the silica content in the reaction. Moreover, the other variables observed to have significant influence include the morphology and different variations of diatomite frustules or amorphous silica applied and the inclusion of fine-grained biogenic calcite [3]. Another report showed the blended diatomite cement with a specific surface area exceeding 500 m^2/kg has a higher compressive strength in comparison with the OPC [4].

The utilization of diatomaceous earth in high strength and ultra-high-strength concrete mixture has also been studied by several researchers [6-9]. It was discovered that supplementary cementing materials (SCM) containing a high amount of silica (SiO_2) such as diatomaceous earth powder (DEP), fly ash, silica fume reacted with a secondary product from cement hydration in the aggregate mortar interface, $Ca(OH)_2$, to form calcium silicate hydrate (CSH). This makes the concrete to be denser and, consequently, to possess higher strength The use of 10% metakaolin with cement has the best performance of mechanical properties of high strength concrete at high temperatures [10]. It has been reported that DEP from Aceh Besar district - Indonesia contains 56.00% silica and fulfills the requirement for SCM [11]. The use of the DEP as cement replacement up to 40% in mortar production resulted in high compressive strength of mortar [12]. The compressive and flexural strengths of blended cement with diatomite have been previously presented [13]. However, the microstructure of composite cement pastes with DEP from Aceh Besar district - Indonesia has not been studied. Therefore, the purpose of this research was to evaluate the setting time, water demand, flow, compressive strength, and especially the microstructure of composite cement paste with DEP from the Aceh Besar district.

2. Materials and methods

2.1 Materials

This study made use of certain materials and they include OPC, diatomaceous earth, and water. The OPC has 3.16 specific gravity, 539.8 m^2/kg specific surface area while its chemical compositions are presented in Table 1. The compact mass of the diatomaceous earth was obtained from Beureunuet Village, Aceh Besar, Sumatra Island, Indonesia (Figure 1). The diatomaceous earth was crushed and calcined at 600 °C for 4 h to have greater reactivity for the pozzolanic activation purpose [14-18]. This process was followed by grinding and sifting with a #200 sieve to produce a powdered form of the material (DEP). The specific gravity, specific surface area, and absorption of DEP were determined based on ASTM C311/C311M-18 and ASTM C1069-09 [19-21]. The test results showed the specific gravity, specific surface area, and absorption rate were 1.88, 675.6 m^2/kg and 6.54 %, respectively. Meanwhile, OPC and DEP particle size distribution are indicated in Figure 2.

Table 1 Chemical composition of OPC and DEP (%).

Chemical analysis	OPC	DEP
SiO_2	17.90	56.00
Al_2O_3	2.32	6.50
Fe_2O_3	4.67	26.40
MgO	2.13	0.00
SO_3	2.56	0.00
CaO	70.34	0.00
K_2O	0.73	0.00
ZrO_2	0.00	7.20
TiO_2	0.00	1.50
Sb_2O_3	0.00	1.30
MnO_2	0.00	0.29
ZnO	0.00	0.27
SnO_2	0.00	0.20
NiO	0.00	0.11
Cu_2O	0.00	0.05
PbO_2	0.00	0.02

The mineralogical characterization and crystal structure of the DEP were studied using the X-ray diffraction (XRD). Moreover, Figure 3 shows the radiation pattern obtained using the XRD tube at 0.154184 wavelengths with the most substantial 3 peaks found to be 27.755°, 26.583°, and 29.901° at 2θ with means that the most significant crystalline minerals of this calcined DEP were silica in the form of quartz, graphite, and lead-silver thallium antimony sulphide. The XRD pattern of DEP was also presented in the previous study [22].

The analysis of the topography or surface texture and morphology which involves the size and shape of the particles for the DEP were conducted using SEM image and the results are indicated in the following Figure 4.

A cylindrical shape having a circular cellular structure with approximately 2 μm diameter was found for the particles. The results for the chemical composition which was determined using the X-ray fluorescence (XRF) are presented in Table 1 with the main chemical composition observed to be SiO_2 and Fe_2O_3.

2.2 Preparation of composite cement paste and determination of water demand

The DEP was blended with OPC to produce composite cement at 0, 10, 20, and 30 percent (w/w). The composite cement was measured to be 650 g and mixed with different quantities of water in the mixer rotated at (450±5) cpm for 30 sec after which it was stopped for a while and rotated again at (285±10) cpm for 60 sec. Moreover, the water demand was determined using the Vicat test based on the EN 196-3 and ASTM C191-01a standards [23,24] and evaluated at the normal consistency of the paste which was achieved when the needle of the Vicat apparatus penetrates to the limit of (10 ± 1) mm below the surface of the paste within 30 sec.

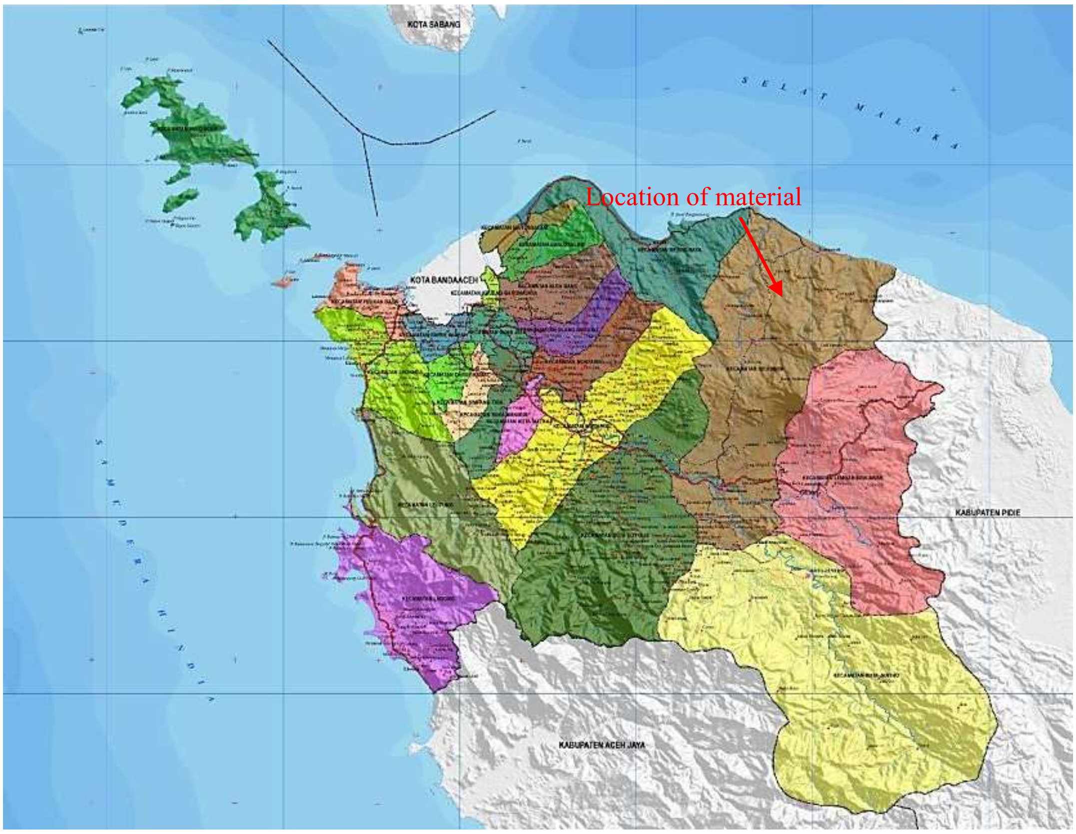

Figure 1 Map of Aceh Besar district.

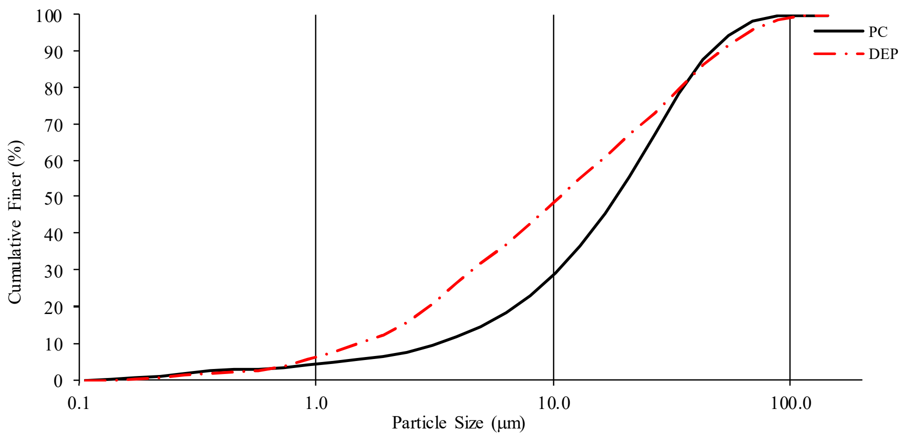

Figure 2 Particle size distributions of OPC and DEP.

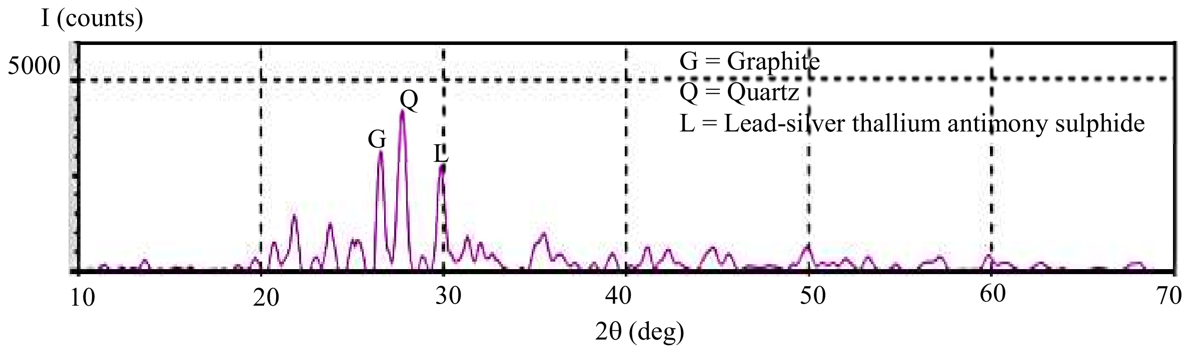

Figure 3 Diatomaceous earth powder XRD pattern.

2.3 Setting time test

The determination of the initial and final setting time for the composite cement paste was based on the EN 196-3 and ASTM C191-01a standards [23,24]. The process involved a gradual mixture of OPC and DEP with water at normally consistent as evaluated using the Vicat test as describe above after which the initial and final setting time were recorded. The time the Vicat needle penetrated 25 mm of the cement paste was recorded as the initial setting time while the time when all the needle length has penetrated was recorded as the final setting time.

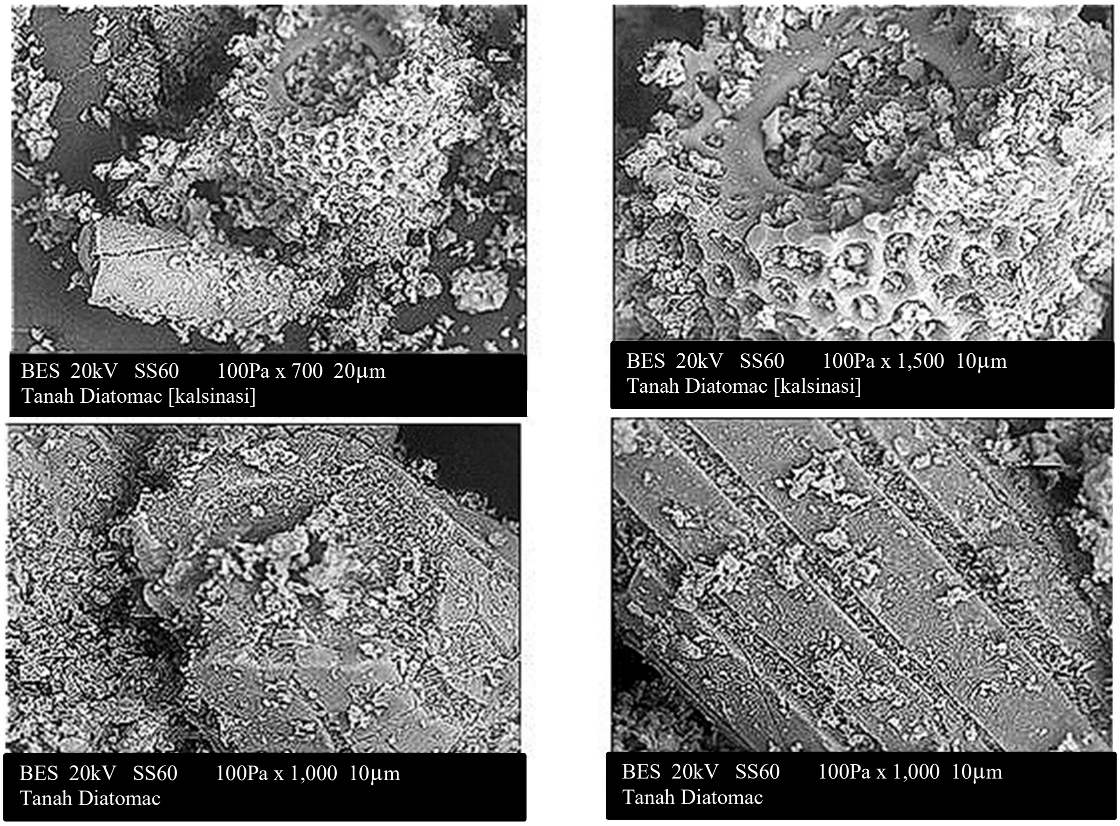

Figure 4 SEM images of DEP.

2.4 Flow test

The composite cement paste flow was evaluated in accordance with the ASTM C1437-07 and C230/C230M-08 [25,26]. The process involved the placement of standard cone at table center and filled with the composite cement paste freshly produced at a thickness estimated to be 25 mm and tamped 25 times. The same process was implemented in the second layer up to when the full cone was achieved. This was followed by the lifting of the cone 1 minute after the completion of the mixing process after which the table was dropped immediately 25 times for a period of 15 sec. The measurement of the mixture flow was conducted in two diameters which are perpendicular.

2.5 Compressive strength test

The measurement of the compressive strength was in line with the standards required by the ASTM C109/C109M-02 [27]. This means the preparation of the 50 x 50 x 50 mm^3 cube specimens involves the casting of the composite cement paste and separation from the mold after 24 h. Moreover, the curing of the specimens was conducted in water for 28 and 120 days after which the compression was tested through the use of a universal testing machine which involved applying load up to the moment the specimens experienced failure.

2.6 Microstructure analysis

The microstructure of the composite cement pastes was analyzed using SEM image on the 120 days old pastes. The sample used for SEM analysis was the remaining specimens used for 120 days compressive strength test. Meanwhile, the XRD test was conducted to analyze the main mineral and crystal structure of the composite cement pastes while their bonding identification was evaluated using Fourier transform infrared (FTIR) test.

3. Results and discussion

3.1 Water demand and setting time

Water demand has been defined as the water quantity required for the preparation of a cement paste at normal consistency based on the standards required in EN 196-3 [23] The water to cement ratio (w/c) for the OPC paste without DEP at normal consistency was found at 0.2463. Table 3 shows water demand increased with the DEP content due to the absorbent nature and fine grain size of DEP. It was also discovered that some of the mixing water was absorbed into DEP at an absorption rate of 6.54% while those required for the hydration process remained constant. This was evident with the use of 30% DEP which led to an increment in water demand by 31.67%. According to Table 3, the initial setting time was observed to have decreased when the DEP content increased. The reduction in the initial setting time was due to the higher specific surface area and fineness of DEP particles compared to OPC as shown in Figure 2. This increased the possibility of cement hydration at an early time, thereby, causing its early plasticity. Similar results have also been reported in some other studies [28,29].

Table 2 Water demand and setting time.

DEP content (% w/w)	Water demand (% w/w)	Setting time (min)	
		Initial	Final
0	24.63	86.06	135
10	26.22	69.65	105
20	29.32	69.28	135
30	32.43	62.60	120

3.2 Flow test results

The flow of composite cement paste was found to have decreased when the DEP content was increased as shown in Table 4. This confirms the ability of the DEP to absorb water into its particles. It was also discovered that the composite cement paste has lower workability in comparison with the OPC paste. Similar results have also been reported in some other studies [2,28].

Table 3 Flow of blended cement paste.

DEP content (% w/w)	Flow of cement paste (%)
0	107.49
10	103.38
20	101.34
30	97.86

3.3 Compressive strength

According to Figure 5(a), the composite cement paste was found to have good compressive strength, such that after 28 days, composite cement with 10%, 20%, and 30% DEP content was found to have reduced by only 2.32%, 7.02%, and 9.26 %, respectively. This was observed to be due to the delay reaction of SiO_2 in DEP with $Ca(OH)_2$ to produce calcium silicate hydrate (CSH) progressively after the paste has passed 56 d and, after 120 days, the reaction produced denser product with increased compressive strength as shown by the results. At the age of 120 days, the compressive strength with 10% DEP content was 2.09% higher than OPC paste while at 20% and 30%, it was 4.29% and 21.60% lower respectively. This is because some DEP particles at 30% content did not react and remain as particles as shown in Figure 7(D) due to the presence of excess silica in the second reaction. Figure 5(B) shows the ratio between the compressive strength of composite cement (f'_{cCC}) to OPC paste (f'_{cOPC}) at the age of 28 days and 120 days.

The relative strength (RS) was defined by Kastis et al [2] as the corrected value of composite cement strength with due consideration for the content of the pure cement and calculated using the following equation:

$$RS = (S \times 100)/C \quad (1)$$

The S represents the composite cement compressive strength while C is the content of pure cement in the composite cement (%) which shows how the DEP added affects the cement hydration. The relative strength (RS) at the 28 and 120 days old presented in Figure 6 shows the composite cement paste has a higher value compared to OPC paste for all DEP content. At the 120 days old, the highest value was attained at 20% content and this means it is the percentage to achieve the best composite cement.

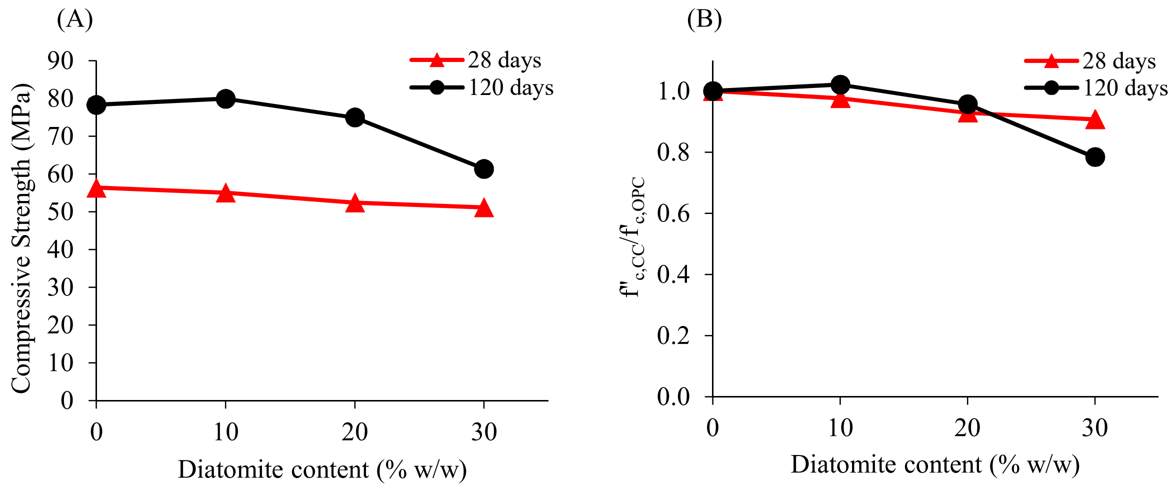

Figure 5 (A) Compressive strength of composite cement paste; (B) Ratio of compressive strength of composite cement paste ($f'_{c,CC}$) to OPC paste ($f'_{c,OPC}$).

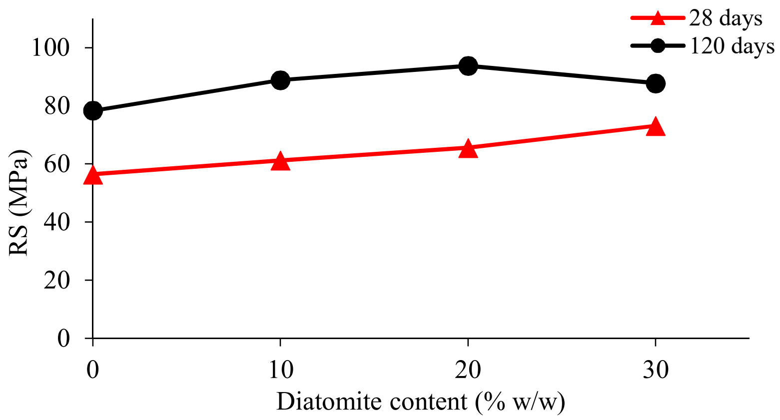

Figure 6 Relative strength (RS).

3.4 Microstructure

Figure 7 shows the SEM images of OPC and composite cement pastes. The CSH product of the OPC hydration is presented in Figure 7(A) with the $Ca(OH)_2$ crystal clearly observed. Moreover, the reaction of the silica from the DEP with $Ca(OH)_2$ to produce CSH is clearly shown in Figures 7(B) and (C). The reduction in the quantity of $Ca(OH)_2$ due to the increase in diatomite content has been reported in a previous study [2]. Meanwhile, Figures 7(B) and (C) show the microstructure of composite cement pastes with 10% and 20% DEP were almost the same with a massive structure of CSH while Figure 7(B) and (C) also show they had a more compact and dense structure compared to OPC paste shown in Figure 7(A). This compact and dense structure was discovered to be the reason for their high resistance to carbonation and aggressive ions [21]. Figure 7(D) shows some DEP particles are in the beginning form without any alteration of the cellular shape due to inactions for the 30% DEP content. Pore structures with an estimated size of 3μm as well as the CSH were observed in Figure 7(D). The CSH was discovered to be developed in the pores as well as on the surface of DEP. Meanwhile, the remaining unreacted DEP particles in composite cement with 30% DEP caused its lower strength as shown in Figure 5.

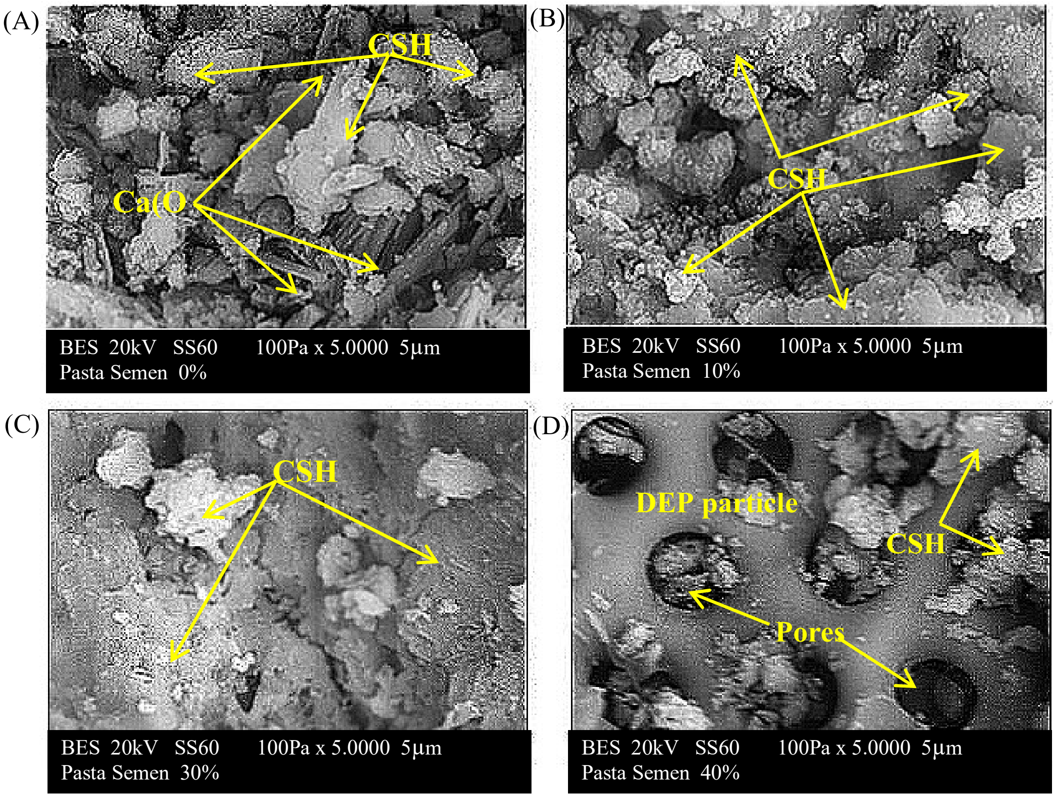

Figure 7 SEM images of composite cement pastes at 120 days with DEP content of: (A) 0%, (B) 10%, (C) 20 %, and (D) 30 %.

Figure 8 shows the XRD pattern for OPC and composite cement pastes and almost similar patterns were observed for OPC and composite cement with 10%, 20%, and 30% DEP. The main products of cement hydration were calcium silicate hydrate (CSH) and $Ca(OH)_2$ with the anhydrous clinker phases almost reacted completely. Moreover, $CaCO_3$ (calcite) was also one of the main crystalline found on the OPC and composite cement pastes due to the ease with which $Ca(OH)_2$ reacts with CO_2 in the atmosphere while orthoclase ($KAlSi_3O_8$) and SiO_2 were also discovered. However, potassium aluminium silicate hydroxide ($KAl_2(Si_3Al)O_{10}(OH,F)_2$) was found in composite cement pastes with DEP but not in OPC paste.

The bonding identification of OPC and composite cement pastes shown in Figure 9 was analyzed based on Syahyadi et al. work [30] and OPC paste was shown to have presented 2 peaks at the wavenumber above 3440 cm^{-1} while all the composite cement pastes presented only 1 peak. This means OPC paste had more stretching of $Ca(OH)_2$ due to the active reaction of silica with $Ca(OH)_2$ in composite cement paste as previously described. This is relevant to the SEM image presented in Figure 7. Moreover, the OPC as well as the composite cement pastes with 10% and 20% DEPs presented 3 peaks at the wavenumber between 1630 cm^{-1} to 3440 cm^{-1} while the composite cement paste with 30% DEP had 2 peaks. This means both pastes had the stretching and bending of H-O-H. Meanwhile, the composite cement pastes with 10% DEP presented 2 peaks while the other cement pastes presented only 1 peak between 880 cm^{-1} and 1140 cm^{-1}. This indicates the composite cement paste with 10% DEP had more stretching of Si-O-Si and Al-O-Si gels in comparison to other cement pastes, thereby, leading to its highest strength as shown in Figure 5. There was, however, no peak recorded between 680 cm^{-1} to 800 cm^{-1} for all cement pastes and this shows the absence of alkali content in them. Furthermore, only the composite cement with 30% DEP presented 1 peak at wavenumbers below 680 cm^{-1}while the others had none and this means the composite cement with 30% DEP had bending Si-O due to its unreacted silica.

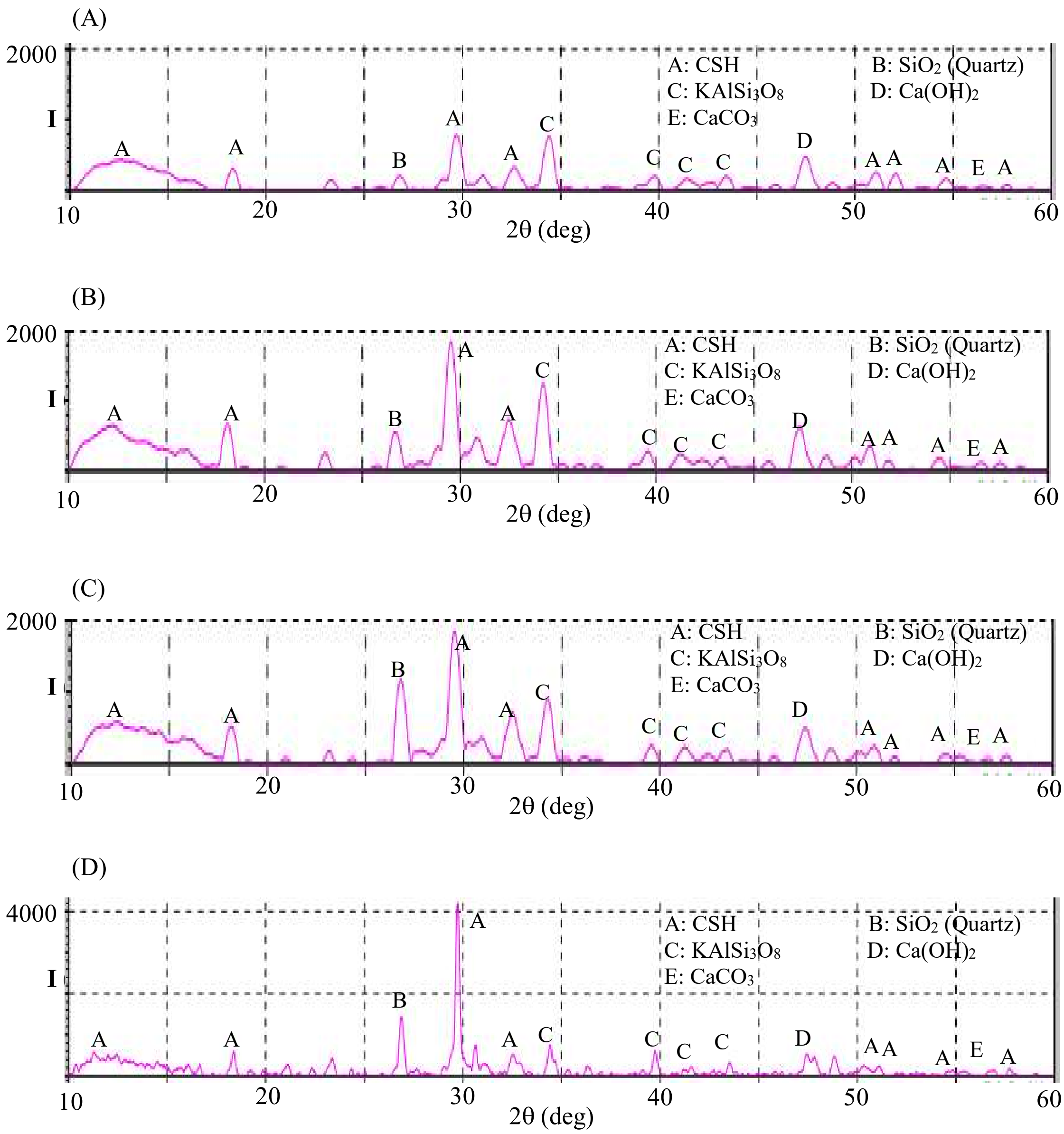

Figure 8 XRD patterns of composite cement pastes after 120 days with DEP content of: (A) 0%, (B) 10%, (C) 20 %, and (D) 30 %.

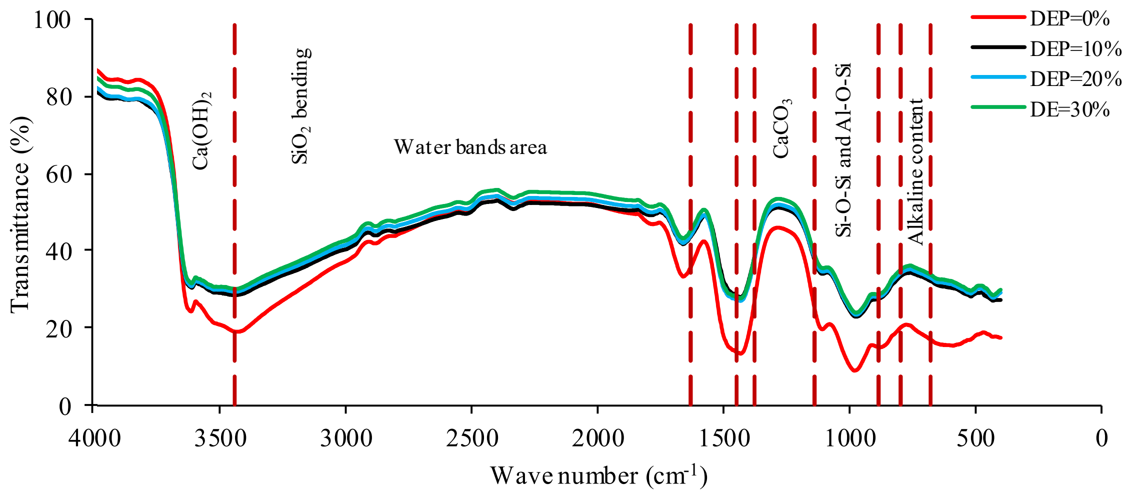

Figure 9 The bonding identification of OPC and composite cement pastes.

4. Conclusion

The study was focused on evaluating the properties and microstructure of composite cement pastes with diatomaceous earth powder (DEP) from Aceh Besar district – Indonesia. The composite cement was produced by blending the Ordinary Portland Cement (OPC) with 0, 10, 20, and 30% content of DEP. It was discovered that the DEP from Aceh Besar District has the potential to be applied as the supplementary cementing material (SCM) to produce composite cement. Its addition to OPC led to a significant increment in the quantity of water demand while a reduction was recorded with the flow of the cement paste. Moreover, the initial setting time of the composite cement was also reduced due to an increase in the DEP content. A microstructural analysis conducted using Scanning Electron Microscopy (SEM) image showed the presence of $Ca(OH)_2$ crystal in the OPC paste while $Ca(OH)_2$ crystal was observed to have reacted with the silica (SiO_2) in DEP to produce calcium silicate hydrate (CSH) in the composite cement pastes with 10% and 20% DEP, thereby, making the paste more compact with higher compressive strength. Meanwhile, the paste with 30% DEP content has some remnant DEP particles which lowered its compressive strength. Furthermore, the XRD pattern also showed the main compounds in the composite cement paste were CSH, $Ca(OH)_2$, $KAlSi_3O_8$, $CaCO_3$, and SiO_2.

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6. Conflict of Interest

The authors state that there is no conflict of interest.

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