Research paper

Strength behavior and microstructural characteristics of soft clay stabilized with cement kiln dust and fly ash residue

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ABSTRACT

This study presents the use of cement kiln dust (CKD) and fly ash (FA) to improve the unconfined compressive strength (UCS) of soft Bangkok clay compared with ordinary Portland cement (OPC). The UCS tests were performed after a curing time of 3, 7, 28 and 90 days. An investigation of each reaction product was conducted using an X-ray diffraction (XRD) technique, and changes in the microstructures of the stabilized clay were observed using a scanning electron microscope (SEM). The test results revealed that a 13% CKD mixture with a partial replacement of 20% FA was suggested as the optimal content ratio to produce a similar long term strength as that achieved using the 10% content of OPC. The UCS of the stabilized clay increased relative to the formation of the primary reaction product, calcium silicate hydrate (CSH), as analyzed using the XRD. The formation of this product reduced the void space in the clay structure resulting in denser and stronger of stabilized clay to correspond with the compressive strength development with time. The change on microstructure of stabilized clay due to the hydration products was evidenced by SEM.

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1. Introduction

Bangkok is located in the Chao-Phraya delta river plain, which is comprised of a soft deposit of marine clay layer, i.e., “soft Bangkok clay”. The undesirable properties of soft clay, i.e., low shear strength and high compressibility, can be strengthened for construction purposes using a process of clay improvement techniques. The most popular clay improvement technique is deep mixing, which is widely used in Europe and Asia (Xu et al., 2006; Xing et al., 2009). The deep mixing technique uses a machine in situ to mix the soft clay and stabilizer to create a clay mixing column to transfer the load to the deep hardened clay layer. The stabilizer commonly used for deep mixing is ordinary Portland cement (OPC), whose increased clay strength is well established (Miura et al., 2001; Mohammad and Alipour, 2012; Yang, 2012). In Thailand, cement is commonly used as a stabilizing agent in geotechnical projects because it is a general construction material that is locally available and can achieve compressive strength within a month.

The mechanism of strength development in clay stabilized with cement (clay cement) can be explained. When cement and water is mixed with clay, primary and secondary reaction products from the hydration reaction are formed, which affect the improved clay cement properties. The primary products are comprised of calcium silicates hydrates (CSH), calcium aluminates hydrates (CAH) and lime. The secondary products resulting from the pozzolanic reaction between lime and clay minerals, clay silica and clay alumina were continuously formed as CSH and CAH after a curing period. As a result of both reaction products, the clay became denser, stronger and harder, which resulted in an increase in the treated clay strength after curing (Bell, 1993; Chang et al., 2007; Ouhadi and Yong, 2008). In addition, cement kiln dust (CKD) is an alternative stabilizer and the mechanism of strength development could also be considered. The CKD is composed of silica, calcium carbonate, calcium sulfate and calcium oxide (free lime), which are minor components of sulfates and chlorides. Since free lime containing binder system reacts with water in the clay, CSH gel is formed due to a pozzolanic reaction and the other products including ettringite, monosulfate and syngenite phase were generated. Thus, the CKD and their hydration products may lead to strength increase of the stabilized clay. Moreover, the combination of CKD and other pozzolanic materials such as slag and fly ash could be more potentially results of mechanical properties (Wang et al., 2004; Peethamparan et al., 2008; Chaunsali and Peethamparan, 2010).

Considering the cost reduction for the deep mixing process, the industrial waste of the alternative stabilizer and the cementitious material was compared with the waste of the OPC. One interesting type of waste is CKD, which is generated in the kiln during the cement manufacturing process. The cement production in Thailand is approximately 30 million tons per year (Thai Cement Manufacturers Association, 2015), and roughly 15–20% becomes CKD (EPA U.S. Environmental Protection...
Fly ash (FA) has long been understood as an industrial waste with pozzolanic materials. Several studies suggested that admixing pozzolanic materials enhanced the long term strength (Kolias et al., 2007; Goodary et al., 2012). The composition of the CKD is highly variable depending on the raw material, dust collection method, operation system and type of fuel used in each facility (Maslehuddin et al., 2009). Based on the above reasons, it is necessary to investigate the CKD before its application in soft clay improvement. A few studies revealed that the use of the CKD as a potential clay stabilizer is comparable to that of the OPC, and it can be used to increase clay strength, reduce permeability and enhance durability of the clay (Peethamparan et al., 2009; Amadi, 2014; Hashad and El-Mashad, 2014).

However, the study shows that the OPC and CKD had a significant increase in the clay strength within a month (Sreekrishnavilasam et al., 2007; Goodary et al., 2012). Several studies suggested that admixing with pozzolanic materials enhanced the long term strength (Kolias et al., 2005; Zentar et al., 2012; Chaunsali and Peethamparan, 2013). Fly ash (FA) has long been understood as an industrial waste with pozzolanic material properties in a concrete field. Fly ash is the industrial waste by-product created from the coal combustion process in power plants that has been pulled out of the boiler by flue gases and collected using electrostatic precipitators into a bag. Generally, the primary chemical composition of FA are silicon dioxide (SiO2), aluminum oxide (Al2O3) and ferric oxide (Fe2O3), hence it can be regarded as a pozzolanic material (Sezer et al., 2006; Wu et al., 2014). In Taiwan, the amount of FA generated from the Mae Moh electric power plant, which is the largest electric power plant, is more than approximately 3.2 million tons per year (Electricity Generating Authority of Thailand, 2015). Using FA would be beneficial for several environmental reasons, such as relieving air pollution, reducing the amount of leachate from FA during storage to seep through the underground water layer and saving natural resources when using FA as a replacement for raw materials used in cement manufacturing process. An increased use of the FA as a partial cement replacement not only adds value of such industrial waste but also presents cost savings in construction projects. A few researchers presented that the FA can be applied in geotechnical projects to enhance clay strength, increase bearing capacity, reduce swell potential of expansive clay, and maintain low permeability of the stabilized clay (Singh et al., 2008; Jongpradist et al., 2010; Kogbara et al., 2013; Voottipruex and Jamsawang, 2014). These studies recommended that a suitable amount of FA should be considered before use due to the variations in its properties at different areas.

The primary objective of the current study is to determine the strength of the stabilized clay using cementitious materials as stabilizers, such as OPC and CKD, and partially replacing the CKD with pozzolanic material using FA. Based on a compressive strength test, the increase in strength of the treated clay was compared with the reaction products from the hydration process, which were investigated using an X-ray diffraction (XRD) analysis. The changes in the stabilized clay structure were observed using a scanning electron microscope (SEM).

2. Experimental program

2.1. Materials

The typical soft Bangkok clay was used as the base clay in this study. The sample was collected from a depth of 2–6 m at a construction site near Bangkok, Thailand. The geotechnical properties of the base clay were determined by the ASTM Standard Test Methods and are summarized in Table 1. The natural water content was 93%, and the liquid limit and the plastic limit was 88% and 36%, respectively, which corresponds to a liquidity index of 1.1. Thus, this clay, when remolded, can be transformed into a viscous form to flow like a liquid. The specific gravity was 2.66, and the undrained shear strengths ranged from 4 to 10 kPa and were obtained from an unconfined compression test. According to the Unified Classification System (USCS), the clay can be classified as clay CH with a high plasticity. The chemical composition of the clay was performed using an X-Ray Fluorescence (XRF) analysis, as indicated in Table 2, to reveal the primary compositions of the clay to be SiO2, Al2O3 and Fe2O3. The stabilizers used in this study were OPC, CKD and FA. The chemical compositions of the stabilizers and the base clay were determined using the XRF analysis, as indicated in Table 2. The specific gravity of the OPC Type I was 3.15. The primary compositions of the commercial OPC Type I were CaO and SiO2 as well as Al2O3 and Fe2O3. The particles of the OPC were observed using an SEM micrograph (Fig. 1a), which illustrates that the particles have a rough surface, sharp corners and are non-uniformly shaped. The CKD was obtained from the cement factory, which is located in the Saraburi province of Thailand. The compositions of the CKD consist primarily of CaO and SiO2 with Al2O3 and Fe2O3, which are similar to those found in the OPC. The CaO and SiO2 contents in the CKD are lower than that in the OPC. The specific gravity of the CKD was 2.73, and the fineness ranged between 3000 and 3400 cm²/g. Fig. 1b illustrates the SEM micrograph of the CKD particles, which revealed that the particles of the CKD were similar to that of the OPC in terms of roughness and sharpness.

The FA used in this study was supplied from the Mae Moh electric power plant, which is located in the Lumphang province of Thailand. The chemical composition of the FA consists of high SiO2 contents with Al2O3, Fe2O3 and CaO. According to the ASTM standard (ASTM C618-12a, 2012), FA is classified as a class C for pozzolanic material properties, for which the composition summations of SiO2 + Al2O3 + Fe2O3 are between 50% and 70% by dry weight. The specific gravity of the FA was 2.53, and the fineness ranged between 3200 and 3600 cm²/g. The SEM micrograph (Fig. 1c) indicated that most of the particle shapes of FA were spherical.

The grain size distribution (GSD) curves for the OPC, CKD and FA were obtained using laser particle size analysis, and the GSD curves for the soft clay were obtained using a hydrometer analysis (ASTM D422-63, 2007). These curves are plotted together for comparison, as shown in Fig. 2. The GSD curves for the OPC, CKD and FA were similar to those of the OPC Class I in terms of roughness and sharpness.

![Table 1](image1.png)

<table>
<thead>
<tr>
<th>Table 1</th>
<th>Geotechnical properties of base clay.</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Properties</strong></td>
<td><strong>Standard test method</strong></td>
</tr>
<tr>
<td>Natural water content (%)</td>
<td>ASTM D2216-10, 2010</td>
</tr>
<tr>
<td>Liquid limit (%)</td>
<td>ASTM D4318, 2010</td>
</tr>
<tr>
<td>Plastic limit (%)</td>
<td>ASTM D4318, 2010</td>
</tr>
<tr>
<td>Plasticity index (%)</td>
<td>ASTM D4318, 2010</td>
</tr>
<tr>
<td>Liquidity index</td>
<td>ASTM D4318, 2010</td>
</tr>
<tr>
<td>Clay content (%)</td>
<td>ASTM D422-63, 2007</td>
</tr>
<tr>
<td>Activity</td>
<td>ASTM D422-63, 2007</td>
</tr>
<tr>
<td>Wet unit weight (kN/m²)</td>
<td>ASTM D7263, 2009</td>
</tr>
<tr>
<td>Specific gravity</td>
<td>ASTM D854-92, 1994</td>
</tr>
<tr>
<td>Undrained shear strength (kPa)</td>
<td>ASTM D2166/D2166M-16, 2016</td>
</tr>
<tr>
<td>Soil classification (USCS)</td>
<td>ASTM D2487-11, 2011</td>
</tr>
</tbody>
</table>

![Table 2](image2.png)

<table>
<thead>
<tr>
<th>Table 2</th>
<th>Chemical composition of base clay, OPC, CKD and FA.</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Compound</strong></td>
<td><strong>Base clay</strong></td>
</tr>
<tr>
<td>Silicon dioxide (SiO2)</td>
<td>61.17</td>
</tr>
<tr>
<td>Aluminum oxide (Al2O3)</td>
<td>21.64</td>
</tr>
<tr>
<td>Ferric oxide (Fe2O3)</td>
<td>3.92</td>
</tr>
<tr>
<td>Calcium oxide (CaO)</td>
<td>1.03</td>
</tr>
<tr>
<td>Magnesium oxide (MgO)</td>
<td>1.68</td>
</tr>
<tr>
<td>Sulfur trioxide (SO3)</td>
<td>1.15</td>
</tr>
<tr>
<td>Potassium oxide (K2O)</td>
<td>2.53</td>
</tr>
<tr>
<td>Sodium oxide (Na2O) + TiO2 + other</td>
<td>0.57</td>
</tr>
<tr>
<td>Loss on ignition (% by mass)</td>
<td>0.91</td>
</tr>
</tbody>
</table>
while the particle sizes of the soft clay were much smaller than those of OPC, CKD and FA. The average of $D_{50}$ (the diameter at which 50% of the particles have a smaller diameter) of the OPC, CKD and FA were 0.01 mm. The base clay is dominated by the clay content with a clay size fraction of 70%, which corresponds to an activity of 0.74.

2.2. Mixture proportions

To improve the UCS of the soft Bangkok clay, the base clay was mixed with the OPC and CKD stabilizers, and the CKD was partially replaced by the FA. Based on the recommendation from a joint study performed in Thailand and Japan (Department of Highways of Thailand and Japan International Cooperation Agency, 1998), the optimum OPC contents generally used in the soft Bangkok clay improvement application range from 80 to 200 kg/m$^3$ of wet clay volume, and the water-cement (W/C) ratios range from 0.8 to 1.2. For example, the high cement contents of 150–200 kg/m$^3$ are frequently used for DCM column applications due to the quality control of the clay-cement mixture (Lai et al., 2006; Horpibulsuk et al., 2011a, 2012a,b; Jamsawang et al., 2011, 2015; Voottipruex et al., 2011a,b). Therefore, in this study, cement contents of 150 and 200 kg/m$^3$ of wet clay volume, which correspond to 10% and 13% of dry clay weight, respectively, were selected, and a partial replacement of the CKD with 10% and 20% FA by dry weight was applied to produce various clay-cement mixtures. The W/B ratio of 0.8 was fixed throughout the experimental program. The details of all the mixture proportions are listed in Table 3.

2.3. Unconfined compressive strength test

The specimen preparation for the unconfined compressive strength (UCS) test was performed in accordance with the JGS standard using the non-compacted method (JGS T821–1990, 1990). The specimens were prepared by pushing the homogeneous clay-cement mixture into a cylindrical mold (50-mm diameter and 100-mm long) and curing for 24 h. After 24 h, the specimens were de-molding and immediately wrapped with a tight plastic sheet to prevent losing moisture content from the specimen surfaces and cured for 3, 7, 28 and 90 days. The UCS tests were performed after the required curing time was reached. The average UCS was obtained from the test results of three samples.

2.4. XRD and SEM analyses

An XRD analysis was performed to investigate the reaction products of the stabilized clay. Furthermore, this method was used to examine the correlation between the UCS and the intensity of the reaction product from the hydration reaction process, which was conducted on the failure plan of the specimen after the UCS test. A SEM analysis was performed to determine the microstructural changes in the stabilized clay.

![Fig. 1. SEM micrographs for: (a) OPC, (b) CKD and (c) FA.](image)

![Fig. 2. GSD curves for base clay, OPC, CKD and FA.](image)

Table 3

<table>
<thead>
<tr>
<th>Sample</th>
<th>Cementitious content (%)</th>
<th>Replacement of CKD by FA (by dry weight), %</th>
</tr>
</thead>
<tbody>
<tr>
<td>OPC</td>
<td>CKD</td>
<td></td>
</tr>
<tr>
<td>C10 (reference)</td>
<td>10</td>
<td>–</td>
</tr>
<tr>
<td>K10</td>
<td>–</td>
<td>10</td>
</tr>
<tr>
<td>K10FA10</td>
<td>–</td>
<td>10</td>
</tr>
<tr>
<td>K10FA20</td>
<td>–</td>
<td>10</td>
</tr>
<tr>
<td>K13</td>
<td>–</td>
<td>13</td>
</tr>
<tr>
<td>K13FA10</td>
<td>–</td>
<td>13</td>
</tr>
<tr>
<td>K13FA20</td>
<td>–</td>
<td>13</td>
</tr>
</tbody>
</table>
used to further investigate the changes in the microstructure of the stabilized clay. After the UCS test, a SEM was used to observe with the same failure plans of the samples those investigated using the XRD analysis.

3. Results and discussions

3.1. Strength development of the stabilized clay

The UCSs for C10 at a curing time of 3, 7, 14, 28 and 90 days were used as a reference to determine the effectiveness of both pozzolanic materials (CKD and FA) in this study. Fig. 3 presents the strength development for all of the stabilized clay specimens. The results reveal that the strengths of the stabilized clay for all of the mixtures increased with curing time, which was expected. The reference UCSs for C10 exhibited the greatest strengths within a month compared to the other mixtures. The UCS of C10 was found to be 317 kPa at 3 days and increased rapidly to 508 kPa and 837 kPa at 7 and 28 days, respectively. The UCS slightly increased to 915 kPa at 90 days. It is observed that the strength characteristics of C10 rapidly increase in the short term, i.e., 3 days and 7 days, continuously increase at 28 days, and slightly increase in the long term, i.e., 90 days.

The UCSs for K10 were 265, 398, 675 and 706 kPa for curing days of 3, 7, 28 and 90, respectively. The UCS developments for K10 and C10 were similar. However, the UCSs of K10 were significantly lower than that of C10. After the partial replacement of the 10% CKD by 10% FA (K10FA10), the UCS also progressively increased with curing time. At 3, 7 and 28 days, the short term strengths rapidly increased; they were lower than that of K10 but slightly higher in the long term, i.e., 90 days. When the CKD was replaced by 20% FA, the UCS of K10FA20 still increased with time. The short term UCSs at 3, 7 and 28 days were relatively lower than those of K10 and K10FA10. Additionally, the UCS of K10FA20 at 90 days was slightly higher than those of K10 and K10FA10 by approximately 3% and 4%, respectively. From the strength development characteristic curve, it can be observed that the short term strengths of the stabilized clay tend to decrease with decreasing CKD content (increase in FA content) while the long term strengths increase with increasing FA content. However, the UCSs of the stabilized clay with CKD-only and CKD substituted partially with FA are lower than that of the OPC stabilized clay for all curing times.

For a higher content of the 13% CKD mixture, the strength of the CKD-only for K13 was increased to 298, 453, 754 and 772 kPa at 3, 7, 28 and 90 days of curing, respectively. The strength of K13 rapidly increased at 3 and 7 days, continuously increased at 28 days and slightly increased at 90 days, which was similar to the strength characteristics of the C10 and K10 mixtures (no FA content). Once the CKD was partially replaced by 10% FA, the short term strength of K13FA10 increased with time but at a relatively lower rate than that of K13. At 90 days, a slightly greater strength than that of K13 was observed. After adding a 20% replacement of FA, the strength of K13FA20 increased with time; however, it was lower than those of K13 and K13FA10 at 3, 7 and 28 days. At 90 days, the strength of K13 and K13FA10 was 9% and 19%, respectively. It can be observed that the short term strength increased rapidly when the CKD content increased while the long term strength tended to increase with an increase in the FA content. The strength development curves have the same characteristic curve as the 10% CKD mixture but with relatively higher strength. K13FA20 exhibits a yield strength that is higher than the C10 reference strength at 90 days of curing time. Thus, K13FA20 is suggested for the optimum content used in this study. Based on the compressive strength test results, it is observed that the CKD has the potential to be used as a cementitious material to enhance the stabilized clay strength. Based on the strength development characteristic curve, the mixture contents of only OPC and CKD have a significant effect on increasing the short term strength of the stabilized clay while those with the FA take a major responsibility to increase in strength in the long term.

Fig. 3 presents the UCS of the stabilized clay for the CKD and the CKD partially replaced with FA at 3 days and 28 days. Similar results from previous studies are also plotted together to compare the strength characteristic of the new material and other stabilized clays with different pozzolanic materials. The correlation of the UCSs at 3 and 28 days was determined using a fitting curve as a linear regression equation as follows:

\[ \text{UCS}_{3d} = 0.398 \times \text{UCS}_{28d} \quad \text{for cement and pozzolan stabilized clay (1)} \]

\[ \text{UCS}_{3d} = 0.376 \times \text{UCS}_{28d} \quad \text{for CKD and FA stabilized clay (2)} \]

Eq. (1) indicates that the UCS of the cement and the pozzolan stabilized clay at 3 days is approximately 0.398 times the UCS at 28 days. For the CKD and the FA stabilized clay, the 3-day strength is approximately 0.376 times that of the UCS at 28 days, as indicated in Eq. (2). It is revealed that the strength of the CKD and FA stabilized clay in this study is slightly less than that of common cement and pozzolan stabilized clay in the early stages of curing. In a similar analysis, the UCS of the CKD and the CKD with FA stabilized clay at 7 days and 28 days curing is compared with the different stabilized clay with cement and pozzolanic materials, as illustrated in Fig. 4b. The curve fitting by linear regression equation for the strength at 7 days and 28 days can be given as follows:

\[ \text{UCS}_{7d} = 0.626 \times \text{UCS}_{28d} \quad \text{Cement and pozzolan stabilized clay (3)} \]

\[ \text{UCS}_{7d} = 0.587 \times \text{UCS}_{28d} \quad \text{CKD and FA stabilized clay (4)} \]

Eq. (3) indicates that the UCS of the cement and the pozzolan stabilized clay at 7 days is approximately 0.626 times the UCS at 28 days, and Eq. (4) indicates that the 7-day strength of the CKD and the FA stabilized clay is approximately 0.587 times the UCS at 28 days. It is indicated that the increase in the strength of the CKD and FA stabilized clay is slightly lower than that of common cement and pozzolan stabilized clay within a week of curing. Additionally, Horpibulsuk et al. (2011b) present the general theory equation for the strength development of cement admixed with soft Bangkok clay in Eq. (5) as follows:

\[ \frac{\text{UCS}_d}{\text{UCS}_{28d}} = 0.026 + 0.293 \ln(d) \quad \text{(5)} \]

Here \( \text{UCS}_d \) is the strength after curing for \( d \) days; \( \text{UCS}_{28d} \) is the strength at 28 curing days; and \( d \) is the curing time in days. This equation is suitable because hydration is the primary chemical reaction while the pozzolanic reaction is minor. Fig. 4c depicts the strength development...
The study equation is close to the theory equation for cement admixed with soft Bangkok clay, and the general equation in this study can be expressed in Eq. (6) as follows:

\[
\text{UCS}_{d}/\text{UCS}_{28d} = 0.133 + 0.237 \ln (d) \quad (6)
\]

The development characteristic curve of modulus of elasticity (\(E_{50}\)), as indicated in Fig. 5, has a similar trend to the strength development curve. The reference C10 achieves the highest modulus within a month as compared to the other mixtures. It is shown that the modulus of C10 rapidly increased within a week, continued to increase at 28 days, and then slightly increased at 90 days of long term curing. Considering a 10% CKD mixture content, the modulus of K10 rapidly increased at 3 and 7 days. At 28 days, \(E_{50}\) continuously increased and then slightly increased at 90 days. The development curve modulus of K10 has a similar trend to that of C10 but a relative lower content. After partially replacing CKD by 10% FA, \(E_{50}\) also increased with curing time. The short term modulus at 3, 7 and 28 days increased less than that of K10 but had a slightly greater modulus at 90 days. When the FA replacement was increased to 20%, the modulus of K10FA20 also increased with time. At 3, 7 and 28 days, \(E_{50}\) is relatively less than those of K10 and K10FA10 while the long term modulus is slightly higher than those of K10 and K10FA10 at approximately 11% and 6%, respectively. From the characteristic curve of the \(E_{50}\) development, it can be observed that the short term modulus tends to decrease with a decrease in the CKD content (increase in FA content) while the long term modulus tends to increase with an increase in the percentage of FA replacement. However, the modulus of the stabilized clay using CKD-only and CKD replaced with FA are lower than that of the OPC stabilized clay for all curing times.

For a 13% CKD content, the \(E_{50}\) of the CKD alone of K13 rapidly increased at 3 and 7 days, continuously increased at 28 days and slightly increased at 90 days, which is similar to the \(E_{50}\) development curve of C10 and K10 (no FA content). As a result of the partial replacement of CKD by 10% FA, the short term modulus of K13FA10 also increased with curing time but was relatively lower than that of K13. For the long curing time of 90 days, the \(E_{50}\) was slightly greater than that of K13. After a 20% replacement of CKD by FA, \(E_{50}\) of K13FA20 increased with time; however, it was relatively lower than those of K13 and K13FA10 for a short term curing time. At 90 days, the \(E_{50}\) was greater than those of K13 and K13FA10 at 28% and 22%, respectively. It can be observed that the short term \(E_{50}\) increased rapidly when the CKD content increased while the long term \(E_{50}\) tended to increase with an increase in the FA content. These \(E_{50}\) development curves are similar to the development characteristic curve of the CKD and the FA stabilized clay compared with the above theory.
the characteristic curve of the 10% CKD mixture but have a relatively higher $E_50$. The relationships of the $E_50$ and the UCS for the different stabilized clay are illustrated in Fig. 6. The ratios of the $E_50$ to the UCS for the different cement and pozzolanic materials stabilized clay range from approximately 99–159. The data ratio of the stabilized clay in this study was also plotted, and it was revealed that a ratio of approximately 114 is within the range for common cement stabilized clay.

3.2. Investigation on reaction products using XRD analysis compared to strength development

XRD diffractometer measurement was performed to evaluate clay mineral of untreated base clay and reaction produced after admixed with stabilizer, OPC, CKD and FA, and then scanned with a 2Theta value ranging from 2° to 70°. The investigated focusing on the phase change analysis of the products of stabilized clay compared with the compressive strength tested with times. For a better characterization of clay minerals in the base clay sample, the XRD analysis was performed in normal, glycoled and calcined forms (Gapak et al., 2017). In the glycoled form, the base clay sample was ethylene glycole solvated in vapors at 60 °C in an incubator for 72 h for identification of clay mineral phase. In the calcined form the base clay samples was heated to 600 °C in muffle furnace and then cooled at the room temperature before carrying out the XRD to predict change in the $d_{001}$-value of various phyllosilicates. The XRD analysis results, as shown in Fig. 7, present the non-clay mineral (quartz) and three reflections of clay mineral including montmorillonite, illite and kaolinite. The $d_{001}$-values are tabulated in Table 4. The basal spacing at reflecctions for montmorillonite within the range of $d_{001}$-values of 12.5 Å (7.07°, 2Theta) to 15 Å (5.89°, 2Theta) exhibits the presence of Na-montmorillonite ($d_{001}$-value of 12.5 Å) and the Ca-montmorillonite ($d_{001}$-value of 14–15 Å) that corresponds to the previous studies by Peethamparan et al. (2009) and Al-Mukhtar et al. (2010). After glycoled, $d$-value was left shift and increased to 17.95 Å (4.92°, 2Theta). For heating process, the montmorillonite reflection decreased and right shift to a maximum $d_{001}$-value of 11 Å (8.04°, 2Theta). The $d_{001}$-value for illite was 9.96–10.1 Å (8.88°–8.75°, 2Theta), which is constant after ethylene glycole solvated and heated at 550 °C. The $d_{001}$-value for kaolinite was 7.14 Å (12.40°, 2Theta) after ethylene glycole solvated. The kaolinite reflections disappeared after heated at 550 °C.

For XRD analysis of treated clay, K13FA20 was suggested as the optimal mixture content was selected to compare the reaction products of the OPC-only and the CKD-only mixture, i.e., C10 and K10. The XRD patterns of the C10 at 3, 7, 28 and 90 days compared with base clay was presented in Fig. 8a. After cement was added 3 days, it can be seen that the new reflections of calcium silicate hydrate (CSH, $\text{Ca}_6\text{Si}_3\text{O}_{18}\text{H}_2$), calcium hydroxide (CH, Ca(OH)$_2$) and ettringite ($\text{Ca}_6\text{Al}_2(\text{SO}_4)_3(\text{OH})_{12}\cdot26\text{H}_2\text{O}$) phases appeared in stabilized clay. Furthermore, the remaining part of the unhydrated reactant formed tricalcium silicate ($\text{C}_3\text{S}$, Ca$_3$SiO$_5$) and dicalcium silicate ($\text{C}_2\text{S}$, Ca$_2$SiO$_4$) also were found. The XRD pattern indicated that the reflection of the CSH intensity increased rapidly at 3 and 7 days, continuously increased at 28 days and slightly increased at 90 days, which is agreeable to the UCS development curve. The new ettringite reflections also appeared at 3, 7, 28 and 90 days have trend to increase with time similar to an increase of CSH. It is note that ettringite crystal beneficial to enhance concrete strength was suggested by previous study (Xu et al., 2012; Scholzová et al., 2015). The product CH, which has a lower intensity than that of CSH, tends to slightly increase with time, while the reactants $\text{C}_3\text{S}$ and $\text{C}_2\text{S}$ slightly decrease with time. It can be seen that the intensities of $\text{C}_3\text{S}$ and $\text{C}_2\text{S}$ decreased with time due to the increase in the hydration process, thus creating increased amounts of the CSH and CH products during the curing period (Mendoza et al., 2015). In addition, it was also observed that the montmorillonite reflection of the base clay decreased after admixed with OPC for 3 days. The reflection reduction was more pronounced in the case of curing days of 7, 28 and 90. The result of this XRD analysis indicates that the significant reduction of montmorillonite reflection had an effect on changing the mineralogy of the clay as a result of the new major reaction products, CSH and ettringite were formed (Dafalla and Mutaz, 2012). Due to an increasing of CSH development with time and ettringite exhibited yield intensity greater than that of the other products, it is believed that CSH and ettringite are principally responsible for the strength development of the stabilized clay. Fig. 9 was presented the development curve of CSH/Quartz ratio with time which then similar trend as the strength development curve. Fig. 8b presents the XRD pattern of the K10 sample. It is revealed that the CSH and CH products formed were similar to those found in the C10
sample whereas the ettringite was not been found. The developments of the CSH intensity for K10 tend to increase with curing time. It is showed that the CSH intensity increases rapidly in the short curing period of 3 days and 7 days with a continuous increase at 28 days and slightly increases in the long term, i.e., 90 days. The montmorillonite reflection decreased with an increased time, which is similar to C10. It can be inferred that montmorillonite reacts with CKD to generate CSH. The development of the CSH/Quartz ratio has a similar trend to that of the C10 strength development curve but relative lower as showed in Fig. 9. Although K10 has the same content as C10, the CSH product was formed in smaller amounts due to the effect of the CKD having a lower CaO content (Table 2), which is an important composition of the raw material used for the cement hydration process, than that of the cement, as mentioned in a previous study (Chew et al., 2004; Ganesan et al., 2008; Saeed et al., 2014). In addition, non-appeared of ettringite crystal was effected to low strength when relative compared with cement. However, the CKD-only mixture was found to have the same reaction products as the OPC mixture, which can contribute to the increase in strength in the stabilized clay. It is indicated that the CKD can be potentially used as a cementitious material in soft clay improvement.

The XRD pattern of K13FA20 is illustrated in Fig. 8c. The pattern presents CSH and CH as the primary reaction products, similar to those found in the C10 and K10 samples. Furthermore, the ettringite was not found. It was also observed that the montmorillonite reflection reduced with increased time. Consideration of the CSH/Quartz ratio, the lower compared to those of C10 and K10 was presented at 3, 7 and 28 days. For long term curing, i.e., 90 days, CSH/Quartz ratio is higher than that of K10 and is markedly higher than that of the C10 reference mixture as showed in Fig. 9. Although ettringite was not been found in this mixture, the CSH/Quartz ratio were higher than OPC-only and CKD-only for long time curing. It can be believed that the beneficial of
the FA, having pozzolanic material properties, has an important effect on the continuous increase in the amount of reaction products (secondary reaction product) generated for the long term curing period (Moraes et al., 2015). The pozzolanic reaction from the FA was beneficial in contributing to a higher CSH/Quartz ratio consequence relative to the higher stabilized clay strength. The analysis of the CSH formation by XRD was agreeable with the result from the UCS test.

Based on the XRD analysis result, it was observed that the reaction products of the CKD-only and the CKD partial replacement with FA were similar to those found in the OPC mixture. The OPC-only and the CKD-only mixtures formed amounts of CSH in the short term, which gradually increased during the long term curing period, while the mixture with the FA content had a higher CSH/Quartz ratio for long term curing. Due to the rate of increase in the CSH/Quartz ratio having a similar trend as the strength development characteristic curve depicted in Fig. 9, it can be concluded that the CKD and OPC have a significant effect on the strength development in the short term, and the FA played an important role for the strength development in the long term of cured.

Furthermore, soaked specimen of stabilized clay was performed to evaluate the short term durability properties. After soaked for 3 days, the UCSs for C10, K10 and K13FA20 were 242, 189 and 185 kPa, which correspond to 76, 71 and 70% of unsoaked strengths, respectively. The XRD patterns of stabilized base clay after soaked for 3 days are presented in Fig. 10. It was found that the kaolinite reflections for all samples were 12.40° (2Theta), which had the same degree as the unsoaked samples. This is evident that the kaolinite has not been dissolved in short curing.

![Fig. 11. SEM micrographs for stabilized clay specimens: (a) C10, (b) K10 and (c) K13FA20 at various curing times.](image)
3.3. Changes in microstructures of stabilized clay using SEM observation

The microstructure of the clay, which was improved by cementitious materials, can be observed by SEM. The technique was performed after the UCS test was completed to observe the change in the microstructure of the stabilized clay. The observation of C10 is depicted in Fig. 11a illustrated that the major hydration reaction products of OPC stabilized clay are CSH and ettringite, confirmed by XRD analysis, was covered on clay surface in the early stages of 3 days and 7 days. The CSH products as presented in the fabric form was spread distribution on clay cluster and filled pore space between clay particles resulting denser in clay structure. The ettringite crystals detected through XRD was also found on clay surface. The needle-like ettringite crystal was formed between clay and CSH fabric consequence in clay packed structure and stiffer. The agreeable observed by Bahmani et al. (2016) found that the hydration products affecting clay denser and stiffer after cement was applied to soft clay. At 28 days, an additional amount of CSH fabric and ettringite crystals was continuously formed over a period of curing to cover the clay surface which makes clay structure stiffer resulting increased in clay strength. For long term curing, i.e., 90 days, intercrossing between clay particle together with CSH and ettringite were continuous formed affected to the improved clay structure become stiffer resulting increased of clay strength with curing time. In addition, Horpibulsuk et al. (2010) stated that the advantage of continuous growth of hydration products was added up the inter-cluster bonding of clay and contain the pore space between clay particles. This changed to the volume of pores that smaller than 0.1 μm is reduced resulting in clay strength increased. It was found that a change on stabilized clay structure was conformed to XRD analysis result and compressive strength test. Change on microstructure of CKD stabilized clay as showed in Fig. 11b. It was found that the major hydration reaction product is CSH which was detected by XRD analysis. At the initial stage of 3 days and 7 days, CSH fabric was formed cover with clay surface and contained in pore space of clay particle resulting in clay denser. However, the needle-like ettringite was not being observed in SEM micrograph which conformed to not be found in XRD analysis. The continuous formed of CSH with time effected to bonding of clay which increased in clay strength at 28 days. For 90 days, growth of CSH was heterogeneous observed to distribution on clay surface consequence increased strength with curing period. Similar study was found by Peethamparan et al. (2008) presented that the hydration products of CKD stabilized clay affected to increase strength with time. The difference compared of microstructure of CKD and OPC stabilized clay was considered. Although the use of CKD affected to enhance clay strength, it was due to the beneficial of only major CSH to fill with pore space creating of clay denser. For OPC stabilized clay, not only the CSH fabric fill with pore space between clay particles but also intercrossing of ettringite crystals together with CSH and clay cluster effected to clay denser and stiffer. By above reason, OPC stabilized clay was significant increased clay strength than CKD in order to compare with the same amount of stabilizer content.

A significant change in the clay structure of CKD and fly ash stabilized clay, K13FA20, is illustrated in Fig. 11c. It was observed that the main hydration reaction product is CSH which was analyzed by XRD similar to CKD-only stabilized clay in Fig. 11b. At 3 days, CSH fabric was formed cover with clay surface similar to those found in the C10 and K10 sample. Furthermore, it was observe that the FA particles were distributed together with the CSH fabric and filled in the void space between clay particles. This effected to reduce the volume void in clay structure resulting in overall denser. At 7 days, more amounts formed of CSH fabric affected to make the clay became denser with an increased with CSH growth and the continuous forms of CSH fabric were observed at 28 days cured. At 90 days, a significant amount of CSH fabric was formed to cover both the clay surface and the FA particle. At this stage, it was assumed that the abundant CSH fabric along with the FA particles embedded in the clay structure that became a clay matrix, and the FA particles filled the void space in the clay structure, which resulted in higher clay strength after curing. The advantage used of FA to cementitious products was suggested as previous studied found that the FA beneficial to reduced void space of clay particle and enhanced clay strength in long term curing (Wang et al., 2013; Shaheen et al., 2014). Based on the SEM observation, it can be concluded that the formation and the progressive growth of the primary reaction products causing the stabilized clay structure to become stiffer and denser than untreated clay, which results in the increase in strength.
after curing. The results from the UCS tests are agreeable with the results from the XRD analysis and SEM micrograph observations.

4. Conclusions

The compressive strength of the soft clay improved when it was stabilized with OPC and CKD, where the CKD was partially replaced with FA. The cementitious OPC led to a rapid increase in strength in the short term curing period and tended to have a gradual increase in strength for the long term curing period. The CKD-only mixture can improve the strength of the soft clay, which has a similar development trend to that of the OPC strength characteristic curve but is lower in compressive strength. After partially replacing the CKD with FA, it is observed that the long term strength increased with an increase in the FA content. In this study, it was suggested to use 13% CKD and an FA replacement of 20% to lead to a greater strength of the stabilized clay at a curing of 90 days. Otherwise, the modulus of elasticity (Es) changed in a similar trend as that of the strength of the stabilized clay.

The XRD analysis results revealed that the primary reaction products, i.e., calcium silicate hydrate (CSH), played an important role in the increase in the clay strength. The rate of increase in the CSH intensity was similar to that of the strength development characteristic curve. Qualitative correlations between the improved strength and the XRD analysis could be explained by observing the changes in the microstructures of the stabilized clay. It was observed that the growth of the primary reaction product CSH resulted in a denser and stiffer clay structure. This led to the increase in strength after curing. The results of the compressive strength test agreed well with the results from the XRD analysis and the SEM observations.

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