Stabilisation of Soft Soil Using Binary Blending of High Calcium Fly Ash and Palm Oil Fuel Ash

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Abstract

Lime and/or Ordinary Portland cement (OPC) are the traditional binders used in soft soil stabilisation. However, their manufacture has a negative impact on the environment. This paper reports the results of experimental work for the optimisation of a binary blended cementitious binder (BBCB) using two types of fly ash as an alternative for use in soft soil stabilisation. The optimum content of the high calcium fly ash (HCFA) was initially determined along with the effect of grinding activation on the performance of HCFA. Subsequently, the effect of palm oil fuel ash (POFA) pozzolanic reactivity on the engineering properties of soft soil, stabilised with HCFA, was investigated by producing different binary mixtures of HCFA and POFA. Based on the Atterberg limits and unconfined compressive strength (UCS) tests, the combination of POFA with HCFA results in a considerably lower plasticity index (PI) and higher compressive strength than those obtained from the soil treated with HCFA alone. Substantial changes in the microstructure and binders of the stabilised soil over curing time were evidenced by SEM imaging and XRD analysis. A solid and coherent

structure was achieved after treatment with BBCB as evidenced by the formation of C-S-H, portlandite and ettringite as well as secondary calcite.

Keywords

Cement-free binary blends; high-calcium fly ash; microstructure; palm oil fuel ash; pozzolanic reactivity; soft soil stabilisation.

1. Introduction

Soil stabilisation was technically introduced several decades ago. It is used to alter undesirable soil properties, to increase shear strength and decrease compressibility, thus meeting engineering specifications for project sites (Venda Oliveira *et al.*, 2011; Kalkan, 2013). Soft soil stabilisation has traditionally been achieved by mixing soft soils with lime, cement, and/or special additives such as pozzolanic materials. Studies involving lime and Ordinary Portland Cement (OPC) as preferred binder materials, report on their ability to bind soil particles to each other, resulting in an improved material (Farouk and Shahien, 2013; Önal, 2014; Modarres and Nosoudy, 2015). However, the manufacture of 1 tonne of OPC consumes 1.5 tonnes of raw materials involving an energy consumption of 5.6 GJ/tonne and CO_2 emissions of approximately 0.9 tonnes. Cement manufacture represents 6% of total global CO_2 emissions, constituting a substantial environmental burden (Song and Chen, 2016; Zhang *et al.*, 2017). An annual growth of 6.95% has been recorded, the highest increase being 9.0% in 2010 and 2011, with a slowdown to 3.0% in 2012 reaching 3.7 billion tonnes. Reflecting a 5% per annum predicted increase in the global cement market (Merchant Reserch & Consulting ltd, 2013), recent data for the manufacture of cement shows tonnage at 4 and 4.3 billion tonnes in 2013 and 2015, respectively (CEM-Bureau, 2015).

Due to a negative environmental impact and the relatively high cost of cement production, researchers have been motivated to create more environmentally friendly and cost-effective materials to replace or reduce the use of OPC in the concrete industry. These materials are generally by-products, or waste materials, most of them fly ashes (Sivrikaya *et al.*, 2014). Fly ashes are most likely to have pozzolanic properties, which by themselves do not have any cementitious properties, but when added to cement, react to boost the hydration process; such materials are classified as class F fly ash (ASTM international, 2003; Lin *et al.*, 2007). Some fly ashes have an adequate free lime content which means they exhibit high hydration reactivity when mixed with water and are classified as class C fly ash. Such fly ashes have been used as cement-based materials to produce new cementitious material which have been used instead of OPC in numerous construction projects (Edeh *et al.*, 2014; Jafer *et al.*, 2015; Dulaimi *et al.*, 2017; Jafer *et al.*, 2018).

Much research has been conducted using waste materials mixed with OPC or lime, to produce new cementitious materials which perform better than OPC or lime alone. These products have been used in a range of diverse construction projects, such as concrete for building, rigid pavements and soft soil stabilisation. In such cases, these waste materials are called supplementary cementitious materials (SCMs). They either have a high amorphous silica content, which facilitates pozzolanic reactivity in the presence of a free lime, such as palm oil fly ash (POFA), rice husk ash, pulverised fuel ash and silica fume (Kumar *et al.*, 2007; Jaturapitakkul *et al.*, 2011), or have a good proportion of free lime, performing as cement when mixed with water, such as ground blast furnace slag, sewage sludge ash, and calcium carbide residue (Fava *et al.*, 2011; Horpibulsuk *et al.*, 2012; Dave *et al.*, 2016).

POFA is a pozzolanic waste material produced from the palm oil industry, generated in huge quantities, mainly in developing countries (Karim et al., 2013). Indonesia and Malaysia are the dominant palm oil production countries, manufacturing 86% of global supplies making them the premier POFA producers (Aprianti, 2017). It was reported by Shafigh et al. (2014)

that Malaysia's production of crude palm oil is 7 million tonnes per annum, a hundred thousand tonnes of POFA per year reported to be produced by Thailand (Jaturapitakkul et al., 2007). The disposal and transportation of the solid waste generated from POFA activity is however a serious problem regarding both the environment and cost making it necessary to address this problem, not only in terms of landfill issues but also increasing construction costs and air pollution.

The effect of the partial replacement of cement by POFA on the compressive strength and sulphate resistance of mortars was investigated by Tangchirapat et al. (2009). Compressive strength with 10% POFA increased by 102% - 104% over that for OPC type I. The use of ground POFA (10-40%) as a cement replacement resulted in a significant reduced rate of mortar expansion in its first year of aging but the compressive strength was less than that for their reference mortars. In the field of soft soil stabilisation, POFA has been used as a cement replacement material in order to improve Atterberg limits and unconfined compressive strength (Ahmad *et al.*, 2011; Pourakbar *et al.*, 2015). However, there are few, if any, investigations of POFA as a potential pozzolanic activator with class C fly ash for use in soft soil stabilisation.

Many researchers have utilised binary, ternary and even quaternary blending systems to produce new cementitious materials from different types of waste materials and fly ashes. Chemical activation methods have been used by some researchers to activate base cementitious materials by adding alkaline and/or pozzolanic materials; others have applied a grinding process using ball mills or mortars for mechanical activation (Antiohos *et al.*, 2007; O'Rourke *et al.*, 2009; Sadique *et al.*, 2012; Dave *et al.*, 2016; Soriano *et al.*, 2016).

This study aims to improve the compressive strength of a soft soil from Hightown, near Liverpool, using a binary-blended cementitious binder (BBCB) consisting of high-calcium fly ash (HCFA) and POFA.

2. Materials and Methods

2.1 Soil samples

The soil used in this study was collected from a site located in Hightown to the north of Liverpool, United Kingdom. The site is a riverbank of the River Alt estuary. The soil samples were extracted from depths ranging between 30 and 50 cm below ground level. The site in general is an alluvial plain; the soil is described as medium-soft, dark grey, silty clay with a trace of sand.

Fig. 1 shows the particle size distribution (obtained from sieve and hydrometer analysis) of the soil used in this study. It contains 13.1% sand, 43.9% silt and 43.0% clay, the main physical and geotechnical properties of the soil are listed in Table 1 along with the results of pH and Loss on Ignition (LOI) tests. An LOI test was performed according to British standard 1377-3 (British Standard, 1999a) by adopting a procedure as explained in clause 4. This test was to determine the organic matter content in the original soil, found to be equal to 7.95%; this value denotes that the soft soil used in this study is considered as medium organic soil according to BS EN ISO 14688-2:2004+A1 (European Committee for Standardization, 2013). Based on BS EN ISO 14688-2:2004+A2013 (European Committee for Standardization, 2013), the grain distribution and Atterberg limits (LL and PI), this soil is classed as an intermediate plasticity silty clay with sand (CI).

Fig. 1. Particle size distribution of the silty clay.

Table 1. Physical and geotechnical properties of the silty clay.

2.2 Binder materials

Two different types of waste material fly ash were used in this study to produce the binary blended cementitious material:

1- High calcium fly ash (HCFA), generated from power plants using an incineration process at temperatures between 850^{o}C and 1100^{o}C by means of a fluidised bed combustion system and;

2- Palm oil fuel ash (POFA), a waste material produced from the incineration processes applied to palm oil fibres at temperatures ranging between 800 and 1000^{o}C, imported from the Sg. Tengi Palm Oil Factory, Kuala Kubu Bharu, Selangor, Malaysia.

2.3 Analytical techniques used for characterisation

2.3.1 Particle size distribution (PSD) of the binder materials

The PSD of the binder materials was obtained using a Laser Diffraction Particle Analyser (LDPA) brand Beckman Coulter LS 13 320. PSD curves of the fly ashes are shown in Fig. 2 alongside that of OPC for comparison purposes. It should be noted that the POFA was sieved first to remove materials which were not completely combusted using a sieve size 150µm. It was then ground with a pestle and mortar grinder for approximately 15 minutes, to increase its fineness and enhance the pozzolanic reactivity (Aprianti *et al.*, 2015). A similar procedure was adopted by Awal and Shehu (2013) for POFA preparation prior to use in experiments. From Fig. 2, it can be seen that the HCFA particles are coarser than those of OPC and POFA. This detail will affect the pozzolanic reactivity of the HCFA; this is discussed in more detail later.

Fig. 2. PSD of the fly ash used in this study in comparison to the PSD of OPC.

2.3.2 Scanning electron microscopic (SEM) analysis

This technique has been used to identify the surface morphological, particle shape and state of the raw materials used in this study along with the microstructures of the hydrated pastes of the treated soil and optimum binary mixture after being exposed for different curing periods. SEM testing was conducted using an FEI Quanta 200 scanning electron microscopy with an accelerating voltage of 10kV, however, the voltage sometimes requires adjustment to below or above 10kV to obtain better resolution. Prior to SEM imaging testing, the specimens were coated with a thin layer of palladium using a sputter coater for increased visibility. The photomicrographs of the binder materials and the virgin soil used in this study are shown in Fig. 3. The flaky-shaped particles which represent the clay in the soil are easily recognised (Fig. 3a). The HCFA particles are agglomerated and have a coagulated state occurring in clusters while POFA has spherical-shaped particles with some irregular-shaped particles with sharp angles as shown in Figs. 3b and c respectively. It was indicated by Segui *et al.* (2012) that the high porosity of binder materials with an agglomerated morphology could lead to a reduction in the workability due to the increase in the water absorbed by the large open areas of high porosity. Chandara *et al.* (2011) reported that POFA contained 67.22% of glassy phase which makes it possible to react as a pozzolanic material.

Fig. 3. SEM photomicrographs of (a) soft soil, (b) HCFA, and (c) POFA.

2.3.3 Energy dispersive X-ray fluorescence (XRF) analysis

The XRF technique was utilised to provide the elemental analysis of the binder materials used in this study along with the virgin soil. The equipment used for this technique was a

Shimadzu’s EDX-720 Energy Dispersive X-Ray Fluorescence Spectrometer. The materials were subjected to the XRF testing in their virgin dry powder state.

The chemical compositions of HCFA, POFA and OPC, are listed in Table 2. From this table, the CaO and SiO_2 contents of HCFA are similar to those for OPC. The CaO content of the HCFA used in this study is comparable to that reported by Dulaimi *et al.* (2016) (67.057%) but is higher than that reported by Sadique and Al-Nageim (2012) at 57.0%. Based on the chemical analysis, HCFA is promising enough to have the potential to form the basis for the cementitious material as it can react with pozzolanic materials to produce cementitious compounds. With respect to POFA, similar chemical properties were reported by Chindaprasirt *et al.* (2014) but with slight differences, specifically in potassium and aluminium oxides (Table 2).

Table 2. The main chemical compositions of the source materials determined by XRF.

2.3.4 X-ray diffraction (XRD) analysis

The XRD analysis was carried out using a Rigaku Miniflex diffractometer with Cu-Kα X-ray radiation at a voltage of 30kV. An electrical current of 15mA was used with a scanning speed of 2.0 degree/min in continuous scan mode, starting from 5^o rising to 65^o. The database released by CDS national chemical database service was used in identifying the peaks. As shown in Fig. 4a, the main mineralogical constituents of the virgin soil are quartz and kaolinite while illite, calcite, potassium feldspar, plagioclase (albite phase), and muscovite are the minor phases. According to Budhu (2011) and Kinuthia (2016), the minerals kaolinite, illite, smectite and montmorillonite are the most common types of crystalline minerals that make up clays. The powder diffraction patterns obtained from the XRD testing of the fly ashes used in this study are shown in Figs. 4b and c. These results reveal that HCFA is crystalline as it has sharp peaks without noticeable noise in the background; this can be observed clearly from Fig. 4b1. The main crystals in HCFA were calcite ($CaCO_3$), lime (CaO), portlandite ($Ca(OH)_2$), and gehlenite ($CaAl[AlSiO_7]$). The reflections indicated at angles 2-theta of 33.3^o and 34^o and 41.2^o are attributed to the presence of tricalcium aluminate ($3CaO.Al_2O_3$) and belite (beta-$2CaO.SiO_2$; larnite) respectively; similar reflections were reported by Gluth *et al.* (2014). Although a similar mineralogy was identified by Dulaimi *et al.* (2017), in the current study, HCFA calcite intensity was found to be higher than that of lime. In contrast, the dominant crystalline peaks of POFA were quartz (SiO_2), while potassium aluminium phosphate ($K_3Al_2[PO_4]_3$) was identified as minor phases (Fig. 4c), similar to the diffraction patterns of the POFA reported by Karim *et al.* (2013). An amorphicity halo has been observed in the XRD patterns of POFA in the angular 2-theta range of 20 – 40^o as shown in Fig. 4c1. Due to the overlapping of the glassy phase of the used POFA, a halo at 2-theta around 22^o is shown; this indicates the existence of a glassy phase and reactivity of POFA (Calligaris *et al.*, 2015).

Fig. 4. XRD pattern of (a) Soil, (b) HCFA, (b1) Scaled HCFA, (c) POFA and (c1) glassy phase of POFA. Where: I: illite; Ka: kaolinite; Q: quartz; P: potassium feldspar; C: calcite; M: muscovite; Pl: plagioclase (albite phase); CH: portlandite; G: gehlenite; L: lime; C_3A: tricalcium aluminate; La; larnite beta-phase; and K: $K_3Al_2[PO_4]_3$.

2.4 Sample preparation

The soft soil exported from the site was first left to air-dry for a few days (not less than 4 days), then oven dried at 110±5^{o}C. The soil lumps were then pulverised using a plastic hammer and sieved on a sieve size 3.36mm.

There are two stages to the experimental work in this research. The first stage was to determine the optimum percentage of HCFA to be added to the stabilised soil and the level of mechanical activation (optimum time of grinding) to be used. Atterberg limits (LL, PL, and PI) determination, compaction parameters (maximum dry density (MDD) and optimum moisture content (OMC)) evaluation and UCS tests were utilised in the first stage. The HCFA was added to the soil for these tests in six different percentages- 0, 3, 6, 9, 12 and 15% by dry weight of the soil. Optimisation of the binary mixture constituted the second stage by mixing POFA with HCFA as a supplementary portion of the optimum percentage of HCFA. The optimum binary mixture was assessed using the UCS test. Atterberg limits and compaction tests were also conducted in the second stage. Finally, and in order to understand the mechanism of improvements in the strength of the stabilised soil, SEM and XRD tests were conducted for the most promising mixtures after 3, 7, and 28 days of curing. Table 3 summarises the testing procedure adopted in this study.

Table 3. The experimental procedure for this study.

The Atterberg limits test (LL, PL and PI) for the untreated and treated soil was conducted in accordance to BS 1377-2:1990 (British Standard, 1998), the LLs determined using the cone penetration method. The standard Proctor test method was used to determine the compaction parameters (MDD and OMC) according to BS 1377-4:1990 (British Standard, 2002).

With respect to the UCS test, a constant volume mould was used to prepare specimens with specific dimensions (38 mm in diameter and 76 mm in height) and densities dependent on the MDD and OMC obtained from the compaction test for each corresponding percentage of HCFA and POFA which was added. This test was performed using a computerised and motorised triaxial machine but without applying any lateral load in the triaxial cell ($\sigma_3 = 0$) in accordance to BS 1377-7:1990 (British Standard, 1999b). Three specimens were prepared for each corresponding percentage of the additive by compressing the soil-binder mixture inside the constant volume mould using a manual hydraulic compression machine. Following this, the specimens were removed from the mould, weighed, wrapped in cling film and labelled, then placed in well-sealed plastic bags, stored for curing at room temperature (20 ± 2^{o}C) for 3, 7, 14, and 28 days. The most promising samples were further cured up to 91 days.

With respect to the samples prepared for the investigation of hydration kinetics (XRD and SEM analysis of the hydrated pastes), at the required curing time, fragments from inside surface were taken and dried at 40^{o}C for 12 hours to stop the hydration, the samples were then subjected to the aforementioned tests without any delay. The fragments for XRD analysis were ground then the powder passed through a sieve size of 150 μm was used, while those for SEM testing were subjected to the procedure explained in section 2.3.2.

3. Results and discussion

3.1 The optimisation of HCFA content

Six different percentages of HCFA - 0, 3, 6, 9, 12, and 15% by dry weight, were added to the soft soil in order to find the optimum percentage of HCFA, as illustrated in Table 3 above. The values of MDD and OMC obtained from the compaction curves of the soil treated with different percentages of HCFA are illustrated in Table 4. It is seen that the HCFA has a significant effect, decreasing the MDD of the stabilised soil. In addition, the OMC increased substantially with the continuous increase of HCFA. However, the results of the compaction test showed that MDD decreased from 1.56 Mg/m^3 for untreated soil to 1.40 Mg/m^3 when adding 15% of HCFA, while OMC increased significantly from 23% to 30.5%. This behaviour is attributed to the high CaO content and the high surface area provided by HCFA, which increased the water demands of the soil-binder mixture (Muhunthan and Sariosseiri, 2008; Jauberthie *et al.*, 2010).

Table 4. Compaction parameters of the soil treated with different percentages of HCFA.

Table 5 shows the Atterberg limits of the soil treated with different percentages of HCFA; LL and PL increased while PI decreased significantly with an increase in HCFA content. The increase in LL was due to an enlargement of the diffuse double layer (expression refers to the floating cations and a few anions around clay particles) caused by an increase in the specific surface area; this in turn increases the water-holding capacity of the soft soil (Das, 2010). There was a constant increase in PL with the increase in HCFA content, an effect that may be due to the cation exchange that occurred between the clay minerals and calcium ions (positive cations) in the HCFA (Eskisar, 2015). Consequently, the PI of the treated soft soil decreased in relation to the increase in HCFA content, this leading to an increase in soil workability, as indicated by Baran *et al.* (2001). Similar behaviour of the Atterberg limits of a soft soil treated with calcium carbide residue (CCR) was reported by Kampala and Horpibulsuk (2013).

Table 5. The effect of HCFA on the consistency limits of the stabilised soil.

The optimum percentage of HCFA required was identified after conducting the UCS tests. Fig. 5a shows the effect of HCFA on the unconfined compressive strength of the soft soil at different curing periods, while Fig. 5b shows the stress-strain diagram for the soil specimens at 28 days of age. The UCS was found to increase with an increase in HCFA content up to 12%, but then decrease at 15% HCFA. The strength of the stabilised soil improved significantly after the first week of curing for all percentages of HCFA. Subsequently, the

UCS increased gradually with respect to 14 and 28 days of curing, as shown in Fig. 5a. The UCS test results indicated that 12% of HCFA was the optimum percentage facilitating the highest values for the compressive strength of the stabilised soil across all curing times.

Fig. 5. (a) The relationship between the UCS and HCFA for different curing periods, and (b) stress-strain diagram for undisturbed, compacted, untreated and soil treated with different percentages of HCFA after 28 days' curing.

The flocculation and agglomeration phenomenon, in addition to the cation exchange that occurred between the monovalent cations of the clay minerals of the stabilised soil and the divalent calcium ions of the HCFA, are responsible for the early strength improvement of the uncured samples (Muhunthan and Sariosseiri, 2008; James and Pandian, 2016). With increases in curing time, the development in the strength of the stabilised soil occurred due to the pozzolanic reaction that took place between the HCFA and the silica and the alumina of the stabilised soil (Aïtcin, 2016; James and Pandian, 2016). This reaction produces cementing materials including calcium-silicate-hydrate and calcium-alumina-hydrate, as shown in Equations 1 and 2:

$$Ca(OH)_2 + SiO_2 \longrightarrow CaO\text{-}SiO_2\text{-}H_2O \qquad (1)$$

$$Ca(OH)_2 + Al_2O_3 \longrightarrow CaO\text{-}Al_2O_3\text{-}H_2O \qquad (2)$$

3.2 Improvement for grinding of HCFA

In order to explore the pozzolanic reactivity of HCFA, the optimum percentage of HCFA (12%) was subjected to four different periods of grinding, namely 10, 20, 30 and 40 minutes. A pestle and mortar grinder with low agitation (1 horsepower = 0.75kW) and a mortar capacity of 2.5 litres was used. It has been shown that applying a grinding technique to fly ash with sufficient lime content and sludge ash, enhances reactivity during hydration and increases the formation rate of calcium-silicate hydrates (C-S-H), this resulting in improved strength and setting properties (Kumar *et al.*, 2008; Zhao *et al.*, 2016). The effect of grinding was established by conducting UCS tests on specimens treated with 12% of HCFA, ground for different periods of time. The curves of particle size distribution shown in Fig. 6 indicated that HCFA ground for only 10 minutes gave the best particle distribution. Longer periods of grinding resulted in coarser grades which were closer to that for unground HCFA due to the agglomeration of HCFA particles. Agglomeration occurs during intensive or longer grinding periods by very strong inter-particle bonds resulting in the formation of a very compacted, irreversible joining of a high number of primary particles (JuhÁSz, 1998). Moreover, the dynamic force of continued grinding causes extra surface energy and surface electric charge which makes the ground particles strongly subjected to attractive inter-particle forces leading to the generation of aggregation and agglomeration (Sajedi and Razak, 2011; Sadique *et al.*, 2013).

Fig. 6. Effect of grinding time on particle size distribution of HCFA.

The results of the UCS tests were in strong agreement with those obtained from the PSD tests, where a higher UCS value was apparent in the soil specimens treated with HCFA ground for 10 minutes, as shown in Fig. 7. This is attributed to the increase in the fineness of HCFA after grinding which boosts the pozzolanic reaction leading to a successful hydration process and forming more cementitious products (Sanjuán *et al.*, 2015). UCS values decreased as the grinding time increased beyond 10 minutes due to the agglomeration which occurred, inversely affecting the pozzolanic reactivity of HCFA and disrupting the hydration process (Sadique *et al.*, 2012; Sadique *et al.*, 2013). Table 6 illustrates the development of UCS over curing time for both the untreated soil and the soil treated with 12% of normal and ground HCFA (Table 3, U4 and GU respectively). The soil treated with activated HCFA achieved higher UCS values over the curing periods. By 28 days of age, UCS had increased by 112.5% after the application of grinding. It should be noted that 12% HCFA was then used as the total binder content for the remaining experimental work in this study.

It was shown that the mechanical activation of additive materials using the grinding technique was successful, leading to an increase in the specific surface area and the production of finer materials which, in turn, enhanced the pozzolanic reactivity of the supplementary cementitious materials (SCMs). Increasing the fineness of binder materials boosts the hydration process increasing the production of cementitious gel, which in turn increases compressive strength (Sanjuán *et al.*, 2015; Velandia *et al.*, 2016). .

Fig. 7. Effect of grinding time on UCS for specimens with 12% HCFA at 7 days of age.

Table 6. Effect of grinding activation (GU) on the development of UCS over curing time.

3.3 Optimisation for the binary mixtures

The optimum binary mixture was identified from the UCS tests conducted on soil specimens treated with 12% of different mixtures of HCFA+POFA, as indicted in Table 3. The effect of binary blending on compaction parameters and consistency limits was also explored in this study. The portions of HCFA used in the binary mixtures were mechanically activated by 10 minutes of grinding, as explained in Table 7. The results for the compaction parameters of the soil treated with the binary blending of HCFA and POFA, are also illustrated in Table 7. It can be seen that MDD increased and OMC decreased gradually with the increase in percentage of POFA. As per the chemical composition of POFA, this fly ash has less CaO content in comparison to that found in HCFA. Thus, the increase in POFA content in the binary mixture decreases the water demand of the binder produced which, in turn, increases the MDD and decreases the OMC. This agrees with studies conducted using fly ash type F in contrast with studies using fly ash type C, which has a higher CaO content (Harichane *et al.*, 2011; Sivrikaya *et al.*, 2014) .

Table 7. Effect of binary blending on compaction parameters of stabilised soil.

The consistency limits of the soil treated with 12% normal and ground HCFA (Table 3, U4 and GU) and different binary mixtures are listed in Table 8. Binary blending is very effective in decreasing the PI of stabilised soil; said PI decreased in parallel with a continuous increase of POFA content in the binary mixture. From Table 8 it can be seen that an increase in the amount of POFA, as a replacement for HCFA in the binary mixture, produces decreases in both LL and PL, the decrease in LL greater than that for PL; PI also then decreases. The decrease in LL and PL is attributed to the decrease in water demand due to a reduction in HCFA from 12% (GU) in the binary mixture as shown in Table 3.

Table 8. Effect of grinding and binary blending on Atterberg limits of the stabilised soil.

Unconfined compressive strength testing was conducted on soil specimens treated with different binary mixtures in order to identify the optimum combination. The specimens were tested after curing periods of 3, 7, 14, and 28 days, the most promising mixtures exposed to a prolonged curing period of 91 days to determine the compressive strength under long term curing. Fig. 8 shows the effect of binary blending on the UCS of the treated soil in comparison to the UCS of the soil treated with HCFA alone at different curing periods. These results indicated that HCFA failed to exhibit substantial pozzolanic reaction in either of its unary mixture states (U4 and GU) when compared with the binders containing POFA, while considerable improvement in UCS was observed with all the binary mixtures. It can be concluded from Fig. 8 that the optimum mixture was BM2, produced by mixing 9% HCFA with 3% POFA by dry weight of the stabilised soil. BM1 and BM3 gave similar results, specifically in the early curing periods.

Fig. 8. UCS development for different mixtures and curing periods.

Over a prolonged curing time (91 days), the UCS of the stabilised soil with mixtures U4, GU and BM2 had significant improvements of 111.16%, 112% and 114.61%, respectively, in comparison to their values at 28 days of curing. The successful development of UCS using binary blending is due to the high pH of both fly ashes used in this study, along with the silica component provided mainly from the glass phase of POFA. The glass phase of silica components dissolves quickly in the alkaline conditions which in turn boosts the chemical activation of HCFA. This leads to a successful hydration reaction, transforming the calcium hydroxide of HCFA into calcium silicate hydrate (C-S-H). Pourakbar *et al.* (2015) reported a similar significant improvement in the UCS of the soil by using 10% binder of binary mixture containing cement-to-POFA proportion of 80:20.

3.4 The kinetics of optimum binary mixture hydration

Fig. 9 shows the phase composition of the dry powder state of BBCB and the comparative diffraction patterns of its hydrated paste at different curing periods. Clear differences can be distinguished in the dry state of the XRD peaks for BBCB and the individual dry states of both types of fly ash. However, the intensities of various peaks are dependent on the participating percentage of each type of fly ash in the binary mixture. In terms of the hydrated states, the amorphous structure of C-S-H and C-A-S-H gels makes it very difficult for them to be identified by XRD (Sadique *et al.*, 2012); such types of hydrates would be reflected in hump forms around 2 theta values 29.5°, 32° and 50° as reported by Kulasuriya *et al.* (2014). However, peaks of different carbonates and hydrates can be distinguished since the early age of hydration (three days) such as calcite ($CaCO_3$), portlandite ($Ca(OH)_2$) and ettringite (Ca_6Al_2 $(SO_4)_3$ $(OH)_{12}$. $26H_2O$). At this age of hydration, the peaks of C3A and larnite (<beta>-C2S) that indicated in the dry powder state of BBCB (non-hydrated) were no longer exist after hydration due to their high reactivity. These compounds were expected to provide the required calcium, silica and alumina along with the silica produced from the dissolution of the glassy phase of POFA to form the C-S-H and C-A-H gel (Gluth *et al.*, 2014). With the progress of curing time, the peak intensities of portlandite decreased and then approximately disappeared while some of ettringite peaks and C-S-H became more pronounced, specifically after 28 days. A progression in the intensities of calcite peaks was also observed due to CO_2 effect on portlandite from air in wet conditions. XRD analysis also revealed a broad peak at 3 days of curing around the angle 2-theta 11.1° suggests the presence of a semi-amorphous C-A-H which became more likely as an amorphous gel after 28 days. A similar finding was reported by Rajamma *et al.* (2009) after XRD analysis of a paste prepared from cement replaced by 30% fly ash. A peak of monocarboaluminate hydrate ($Ca_4Al_2O_6.CO_3.11H_2O$); CO_3-AFm was observed after 7 days of curing at 2-theta 11.7° which developed to be more pronounced after 28 days of hydration. This compound is formed by the chemical reaction between the ettringite and C3A due to the AlO_2^- diffusion which is expected to continue in release even after the sulphate being used up (Peng *et al.*, 2006). The generation of C-S-H gel and CH is attributed to the chemical reaction occurring between the lime (L) and amorphous silica provided by HCFA and POFA, respectively. These compounds are expected to be formed continuously as curing time progresses because the pozzolanic reaction of silica requires a longer period to activate HCFA to form more C-S-H gel (Calligaris *et al.*, 2015;

Jiang *et al.*, 2016). The XRD results show that at the early stages of curing, some peaks of CH developed then decreased at 28 days curing. This indicates that CH was activated with the pozzolanic materials provided by POFA and transformed to C-S-H compounds. Similar findings were reported by Sadique *et al.* (2013) after using a calcium-rich fly ash activated by a high alkali sulphate fly ash.

Fig. 9. XRD patterns of BM2 (BBCB) in dry and hydrated states with different curing periods (CH: portlandite, C-S-H: calcium silicate hydrate; C-A-H: calcium aluminate hydrate; E: ettringite; Q: quartz; L: lime; La: larnite; C: calcite; C_3A: tricalcium aluminate; and Mc: monocarboaluminate hydrate).

The consequent changes in the microstructure of the BBCB pastes, with and without soil, due to hydration over different curing periods, 3, 7, and 28 days, are shown in the SEM test images, Figs. 10a-f respectively. These changes were very clear and represented by the formation of flaky shaped CH crystals and C-S-H gel in addition to needle-shaped crystals (ettringite) which occurred due to the hydration reaction. Ettringite was initially observed at an earlier hydration stage (3 days) with a small amount of CH crystals, as shown in Figs. 10a and d, the subsequent formation of ettringite and CH continuing becoming denser at 7 days curing. At this age, the SEM images showed that some particles of the soil (Fig. 10b) and particles of BBCB (Fig. 10e) were coated with C-S-H gel. Regarding longer curing periods (28 days), uniform and larger amounts of cementitious products were observed, represented by the transformation of most of the CH crystals to C-S-H gel as shown in Figs. 10c and f. Additionally, the rhombohedral structure of calcite crystals was observed clearly in the BBCB paste after 28 days of curing as illustrated in Fig. 10f. The high alkalinity of HCFA and POFA enhanced the dissolution of the glassy phases of HCFA and POFA and produced additional C-S-H gel. This gel tends to fill pores and grow into capillary spaces, resulting in a more impermeable, dense and higher-strength structure (Blanco *et al.*, 2006; Kulasuriya *et al.*, 2014).

When the gypsum source becomes entirely consumed and the C3A continue to dissolve and provide more ions of Ca^{2+} and AlO_2^-, the ettringite starts to react with the newly produced ions and transforms into AFm phase (Marchon and Flatt, 2016). However, longer curing and associated hydration reactions within an alkaline environment were expected to cover the ettringite with a thick layer of C-S-H gel which prevents the diffusion of AlO_2^- and disrupts its reaction with ettringite (Peng *et al.*, 2006).

Fig. 10. SEM images of hydrated pastes of (a) to (c) soil-BBCB and (d) to (f) BM2 (BBCB).

4. Conclusions

The effect of the mechanical activation using low intensity grinding of HCFA, along with the effect of pozzolanic activation using POFA, were analysed in this paper. This provides a novel approach for the use of waste materials in the development of a new binder for use in soft soil stabilisation. According to the results, the following conclusions can be drawn:

- Despite the HCFA having a chemical composition similar to that of OPC, it has less potential as pozzolanic activator than OPC. However, the HCFA has much free lime as well as a small amount of glassy phase. Therefore, it is beneficial to produce a BBCB mixed HCFA with high glassy POFA in order to produce high performance pozzolanic cement for the use in soft soil stabilisation.
- Due to the high water demands of HCFA, MDD decreased and OMC increased with an increase in HCFA content. However, the results of the compaction parameter test indicated slight increments in MDD and reductions in OMC with the use of POFA.

These increments in density contributed to improvements in the strength of the stabilised soil.

- HCFA was found to be very effective in improving the consistency limits of the stabilised soil, where the PI decreased from 20.2 for the untreated soil, to 13.38 by using 12% mechanically activated HCFA. No significant effect was observed using POFA in the binary mixtures, but the PI continued to decrease, dropping to 11.73 for the soil treated with BM4. This reduction in PI will substantially improve the resistance of stabilised soil against swelling and shrinkage stresses.
- Mechanical activation using grinding energy activated the pozzolanic reaction of HCFA by increasing the surface area. However, the results of mechanical activation indicated that long periods of grinding led to agglomeration which caused a reduction in compressive strength due to the decrease in pozzolanic reactivity. Mixing a de-agglomeration material with HCFA during the grinding process would mitigate the effect of extended periods of grinding.
- The results of the UCS tests indicate that the optimum BBCB (BM2) mixture comprises HCFA and POFA at a ratio of 9:3. The performance of this mixture as a binder in soft soil stabilisation was very promising in that soil strength improved significantly from 200 kPa for untreated soil to 1059 kPa for the soil treated with 12% of BBCB at age 90 days; this improvement is 5.7 times the UCS for compacted virgin soil.
- The XRD patterns of the BBCB hydrated paste revealed clear changes in peak intensities over curing time. This explains the consumption of some of BBCB compounds, such as lime and portlandite during the hydration processes, resulting in the formation of new compounds e.g. C-S-H and ettringite in addition to the calcite resulted from the CH carbonation. It may be concluded that the latter compounds contributed to the development of the strength of the soil stabilised with BBCB.

- SEM images for BBCB hydrated paste provided evidence to support the XRD analysis. A sequent formation of C-S-H, CH, calcite and ettringite was observed, over time of curing, using SEM testing which confirms the cementitious property of the BBCB. A similar behaviour was observed in the cured soil-BBCB pastes, a stronger soil structure achieved at 28 days curing.

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HIGHLIGHTS

1. A cement-free binder has been developed for soft soil stabilisation.
2. HCFA significantly contributed to the development of geotechnical properties.
3. The role of grinding, used to activate the pozzolanic reactivity of HCFA, was examined.
4. POFA's pozzolanic reactivity vigorously activated the HCFA.
5. USC development was evidenced through the use of SEM and XRD techniques.

List of abbreviations:

BBCB: binary blended cementitious binder.

HCFA: high calcium fly ash.

LDPA: Laser Diffraction Particle Analyser.

LOI: loss on ignition.

MDD: maximum dry density.

OMC: optimum moisture content.

OPC: Ordinary Portland cement.

POFA: palm oil fuel ash.

PSD: particle size distribution.

SCMs: supplementary cementitious materials.

SEM: scanning electron microscopy.

UCS: unconfined compressive strength.

XRD: X-ray diffraction.

XRF: X-ray fluorescence.

Table 1: Physical and geotechnical properties of the silty clay.

Property	Value
Natural Moisture Content NMC %	36.8
Liquid Limit LL %	44
Plastic Limit PL %	23.8
Plasticity index PI	20.2
Sand %	13.1
Silt %	43.9
Clay %	43
Specific Gravity (Gs)	2.57
γ_{dmax} Mg/m^3	1.57
Optimum moisture content OMC %	23
pH	7.78
Organic Matter Content %	7.95
Unconfined Compressive Strength for undisturbed soil (kPa)	66.46

Mg/m^3= Mega gram/cubic metre.

Table 2. The main chemical compositions of the source materials determined by XRF.

Item	Soft soil	OPC	HCFA	POFA
CaO %	4.485	66.119	66.760	9.047
SiO_2 %	59.884	24.909	25.120	53.011
Al_2O_3 %	7.334	1.728	2.380	6.487
Fe_2O_3 %	3.850	1.668	0.030	4.873
MgO %	2.856	1.322	2.570	1.329
P_2O_5	0.096	-	-	1.794
Na_2O %	1.337	1.357	1.718	1.496
K_2O %	2.960	0.836	0.310	6.501
SO_3 %	0.343	2.655	0.380	2.531

Table 3. The experimental procedure for this study.

Mixture ID	Binder added (%) by dry weight of the stabilised soil		The experiments conducted on the binder-stabilised soil					
	HCFA	POFA	LL	PL	Compaction	UCS	SEM	XRD
UN	0	0	*	*	*	*	*	*
U1	3	0	*	*	*	*	NR	NR
U2	6	0	*	*	*	*	NR	NR
U3	9	0	*	*	*	*	NR	NR
U4	12	0	*	*	*	*	NR	NR
U5	15	0	*	*	*	*	NR	NR
GU	12% ground activated	0	*	*	NR	*	NR	NR
BM1	10.5	1.5	NR	NR	*	*	NR	NR
BM2	9	3	*	*	*	*	*	*
BM3	7.5	4.5	NR	NR	*	*	NR	NR
BM4	6	6	*	*	*	*	NR	NR

UN: refers to the untreated soft soil; U: from unary mixture; GU: for the ground unary mixture; BM: for binary mixture; *: for the conducted experiments; and NR: is not required.

Table 4. Compaction parameters of the soil treated with different percentages of HCFA.

HCFA %	MDD (Mg/m^3)	OMC %
0 (UN)	1.56	23.0
3	1.48	26.0
6	1.46	27.5
9	1.45	28.0
12	1.44	29.0
15	1.40	30.5

Table 5. The effect of HCFA on the consistency limits of the stabilised soil.

HCFA %	LL (%)	PL (%)	PI
0 (UN)	44.0	23.8	20.2
3	47.0	30.4	16.6
6	49.2	34.9	14.3
9	50.5	36.5	14.0
12	51.3	37.8	13.5
15	52.5	39.4	13.1

Table 6. Effect of grinding activation (GU) on the development of UCS over curing time.

Mix. ID	UCS (kPa) at different curing periods (days)			
	Uncured	7	14	28
(UN)	202	206	210	231
U4	367	570	642	690
GU	360	622	725	776

Table 7. Effect of binary blending on compaction parameters of stabilised soil.

Normal HCFA %	Ground HCFA %	POFA %	Mixture ID	MDD (Mg/m^3)	OMC %
0.00	0.00	0.00	Virgin soil	1.56	23.50
12.00	0.00	0.00	U4	1.44	29.00
-	12.00	0.00	GU	1.43	30.00
-	10.50	1.50	BM1	1.45	27.50
-	9.00	3.00	BM2	1.46	26.50
-	7.50	4.50	BM3	1.47	25.50
-	6.00	6.00	BM4	1.48	24.50

Table 8. Effect of grinding and binary blending on Atterberg limits of the stabilised soil.

Mix. ID	LL (%)	PL (%)	PI
UN	44.0	23.8	20.2
U4	51.3	37.8	13.5
GU	52.2	38.8	13.4
BM1	51.4	38.3	13.1
BM2	49.3	36.5	12.8
BM4	48.6	36.5	12.1
BM5	48.0	36.3	11.7

Figure 1

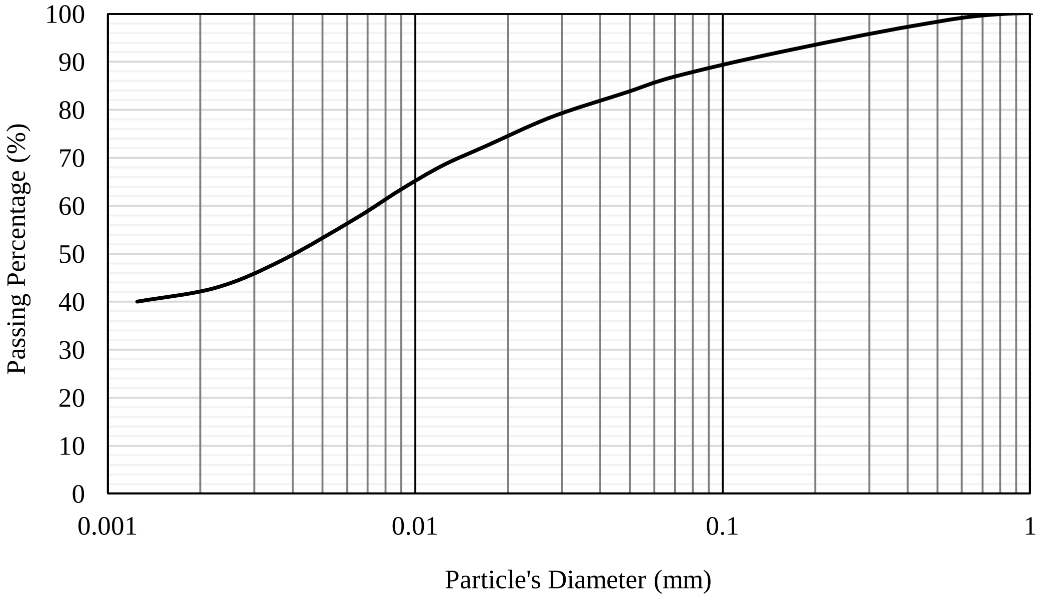

Fig. 1. Particle size distribution of the silty clay.

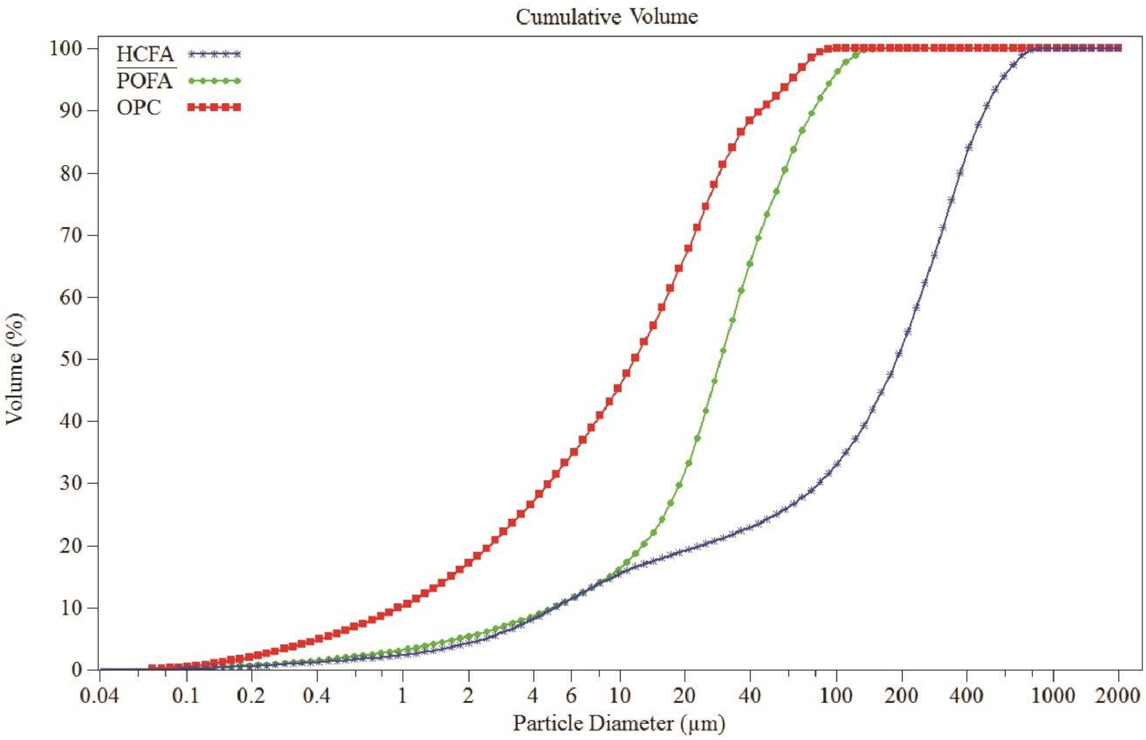

Fig. 2. PSD of the fly ash used in this study in comparison to the PSD of OPC.

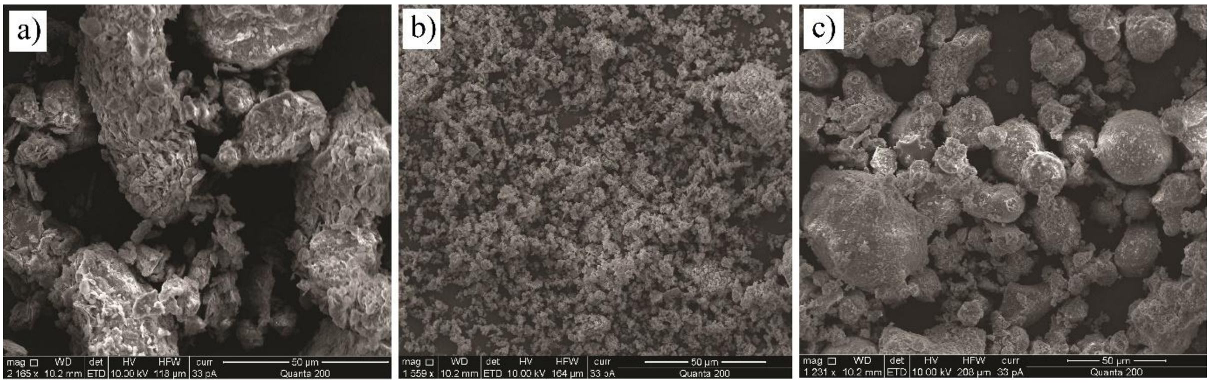

Fig. 3. SEM photomicrographs of (a) soft soil, (b) HCFA, and (c) POFA.

Figure 4

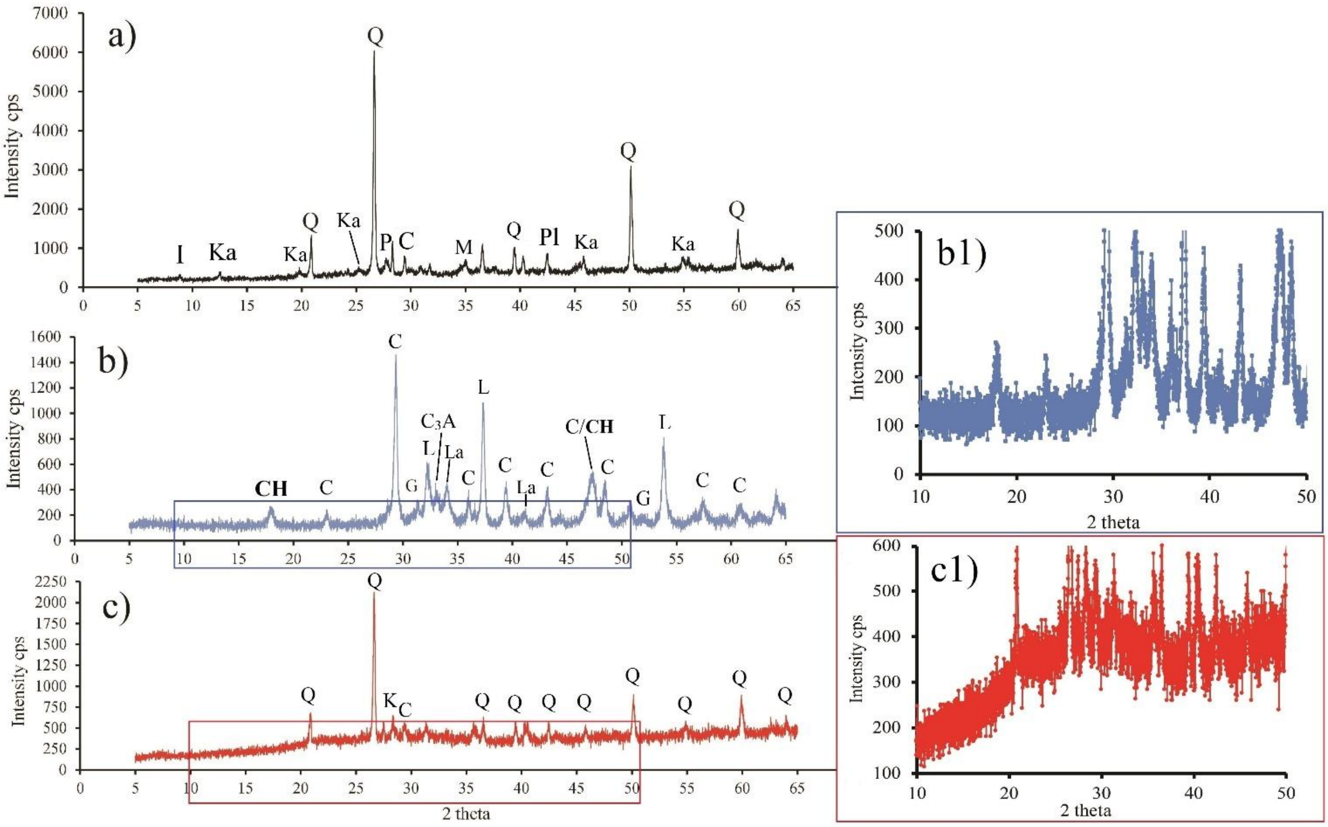

Fig. 4. XRD pattern of (a) Soil, (b) HCFA, (b1) Scaled HCFA, (c) POFA and (c1) glassy phase of POFA. Where: I: illite; Ka: kaolinite; Q: quartz; P: potassium feldspar; C: calcite; M: muscovite; Pl: plagioclase (albite phase); CH: portlandite; G: gehlenite; L: lime; C_3A: tricalcium aluminate; La; larnite beta-phase; and K: $K_3Al_2[PO_4]_3$.

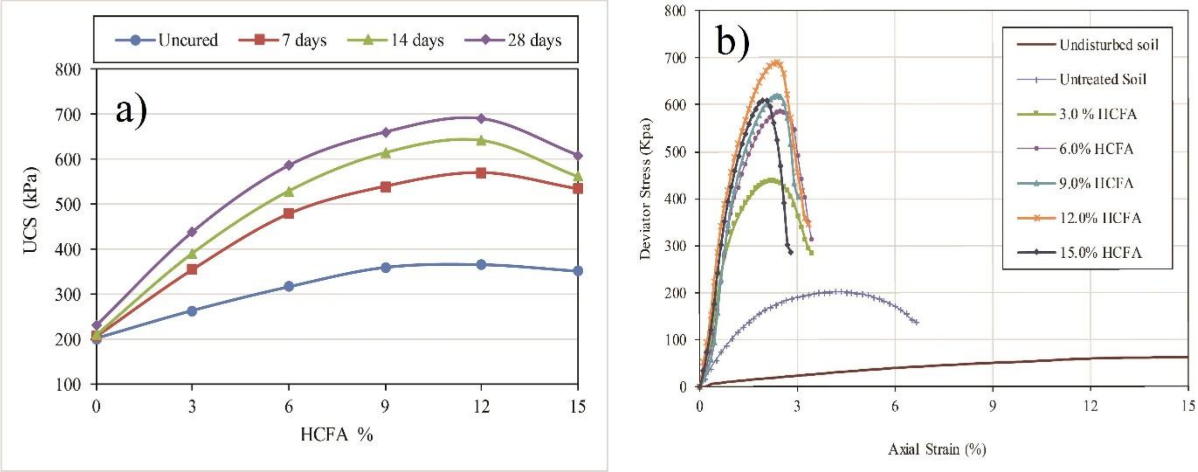

Fig. 5. (a) The relationship between the UCS and HCFA for different curing periods, and (b) stress-strain diagram for undisturbed, compacted, untreated and soil treated with different percentages of HCFA after 28 days' curing.

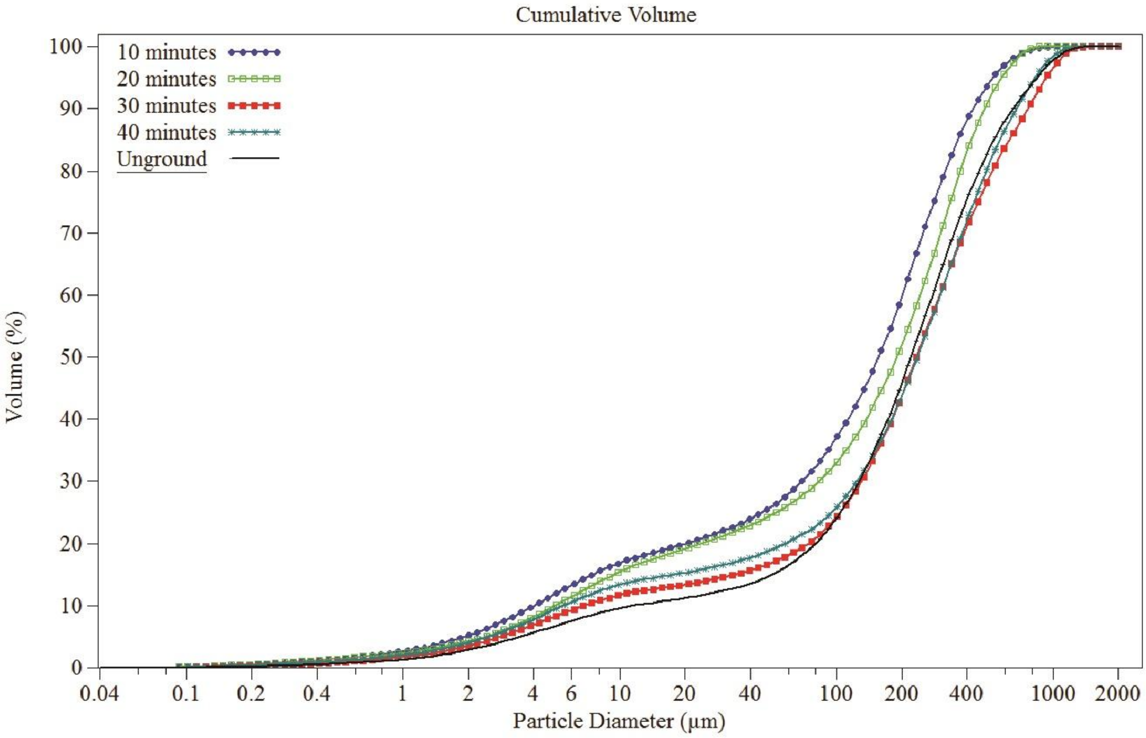

Fig. 6. Effect of grinding time on particle size distribution of HCFA.

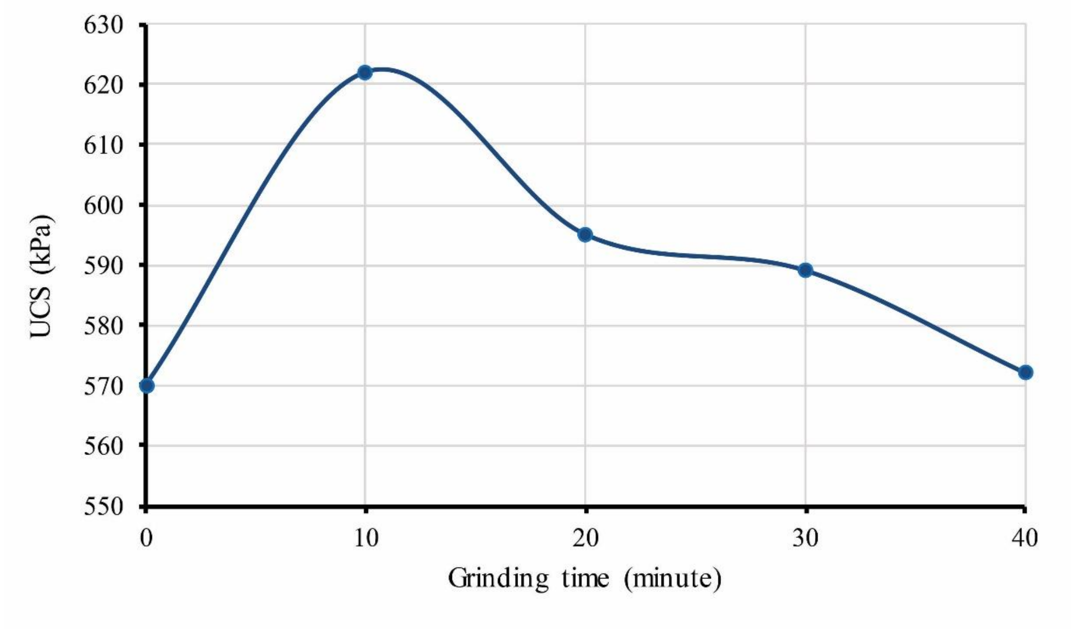

Fig. 7. Effect of grinding time on UCS for specimens with 12% HCFA at 7 days of age.

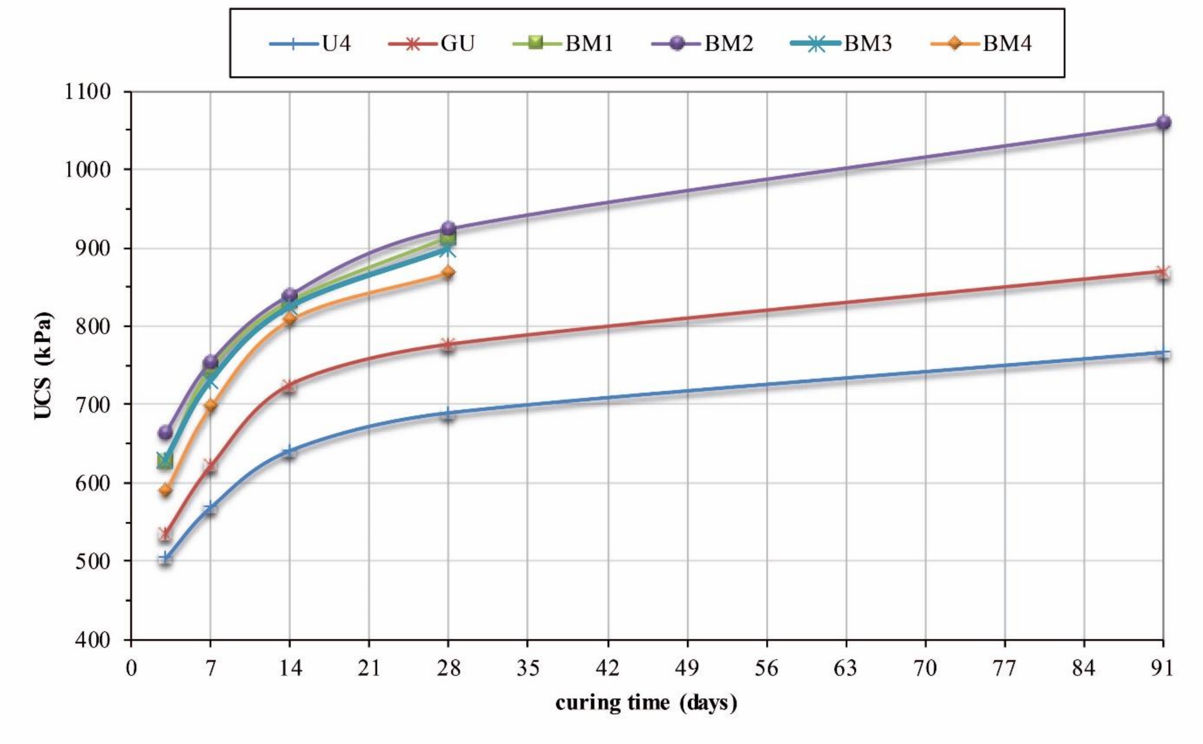

Fig. 8. UCS development for different mixtures and curing periods.

Figure 9

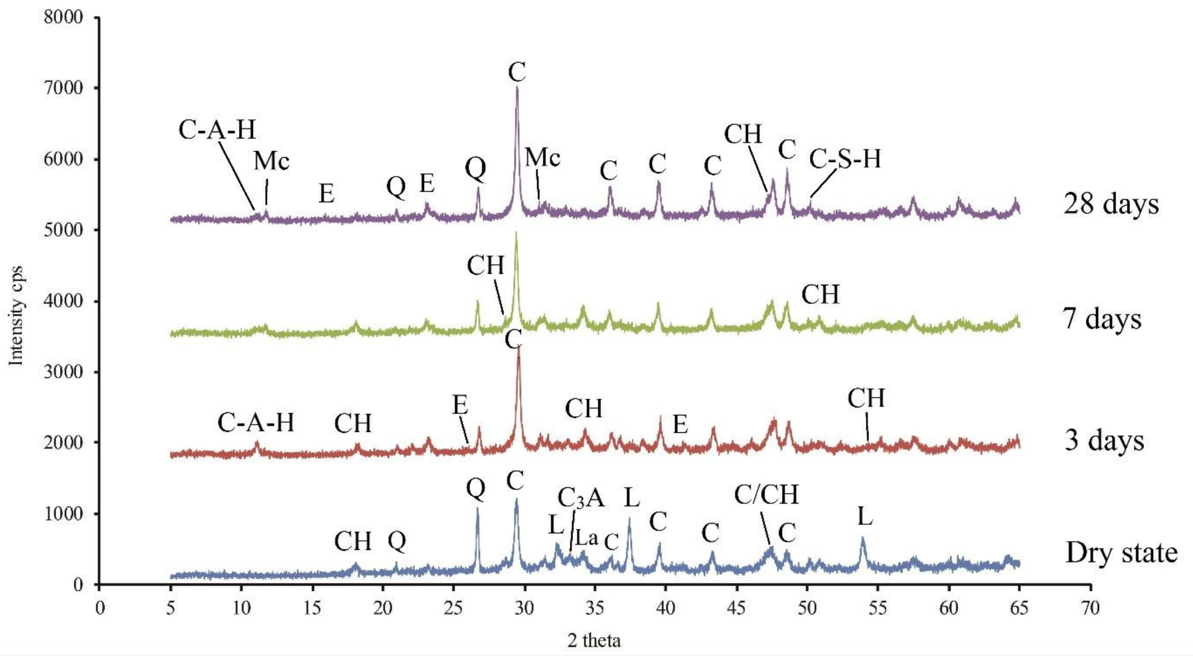

Fig. 9. XRD patterns of BM2 (BBCB) in dry and hydrated states with different curing periods (CH: portlandite, C-S-H: calcium silicate hydrate; C-A-H: calcium aluminate hydrate; E: ettringite; Q: quartz; L: lime; La: larnite; C: calcite; C_3A: tricalcium aluminate; and Mc: monocarboaluminate hydrate).

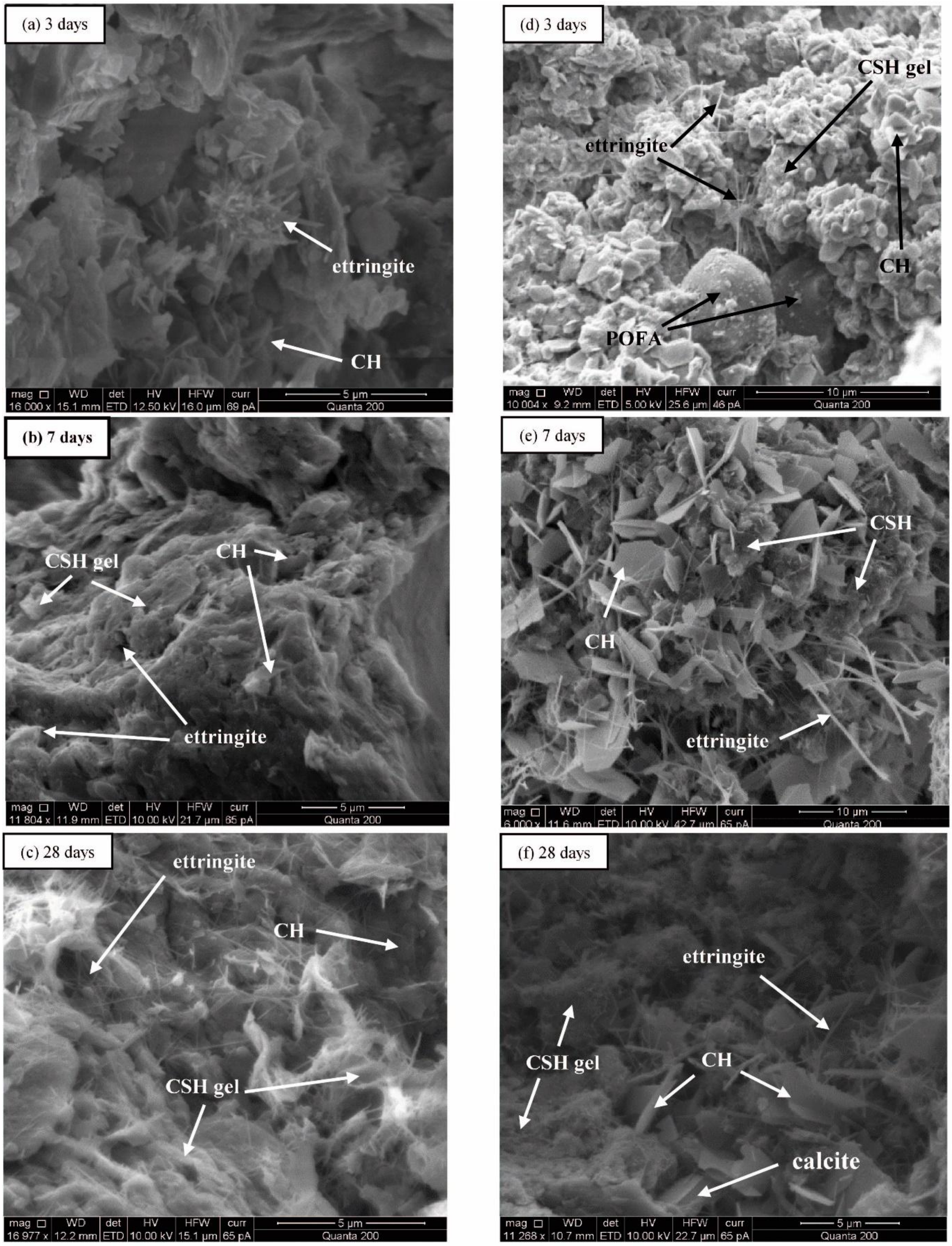

Fig. 10. SEM images of hydrated pastes of (a) to (c) soil-BBCB and (d) to (f) BM2 (BBCB).