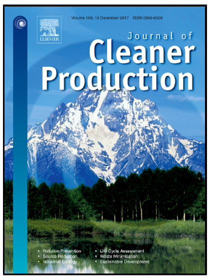

Development of a New Ternary Blended Cementitious Binder Produced from Waste Materials for use in Soft Soil Stabilisation

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HIGHLIGHTS

1. A symbiotic balance of the free lime and silica oxides has been proposed.
2. A cement-free binder has been developed for soft soil stabilisation.
3. FGD gypsum extensively contributed to the compressive strength evolution.
4. UCS evolution was evidenced via the use of XRD and SEM techniques.

Development of a New Ternary Blended Cementitious Binder Produced from Waste Materials for use in Soft Soil Stabilisation

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Abstract

Soil stabilisation using traditional binders such as Ordinary Portland Cement (OPC), has a serious negative environmental impact, specifically carbon dioxide (CO_2) emissions as a result of the manufacture of OPC. Because of this, the use of sustainable binders has become a critical issue to help reduce cement production through the use of by-product materials. This research seeks to develop a new ternary blended cementitious binder (TBCB) to replace cement for soft soil stabilisation. Different ternary mixtures containing wastes i.e., high calcium fly ash (HCFA), palm oil fuel ash (POFA) and rice husk ash (RHA) along with flue gas desulphurisation (FGD) gypsum used as a sulphate activator and grinding agent, were examined. The results illustrate that ternary mixtures improved the engineering and mechanical properties of stabilised soil. The results indicated that the plasticity index (PI) was reduced from 20.2 to 13.0 and the unconfined compressive strength (UCS) increased after 28 days of curing from 202kPa to 944kPa using the optimum non-FGD activated mixture. FGD contributed significantly by increasing the UCS to 1464kPa at 180 days of curing, which surpassed that for the reference cement (1450kPa), and by improving the soil consistency limits; where the PI decreased to 11.7 using TBCB compared with 14.5 for the soil treated with the reference cement. X-ray diffraction (XRD) and scanning electron microscopy (SEM) analysis revealed substantial changes in the diffraction patterns and microstructure components of the TBCB paste over the curing period, confirming the formation of cementitious products. A solid, coherent and compacted structure was achieved after treatment with TBCB as evidenced by the formation of C-S-H, CH and ettringite.

Keywords

By-product materials; FGD gypsum; high-calcium fly ash; microstructure; soft soil stabilisation; sustainable blended binder.

1. Introduction

Climate change and global warming are major risks facing humankind because of their severe effect on the planet. Global warming is linked to the phenomenon of greenhouse gases (GHGs) emissions, CO_2 being the most prevalent of these (Specht *et al.*, 2016). Ordinary Portland Cement (OPC) is one of the most extensively used construction materials worldwide (Karim *et al.*, 2013). It is the most used material after water, having significant advantages as a construction material in different civil engineering industries including soil stabilisation. However, the use of cement has many drawbacks, specifically negative environmental impacts, something which has become a major concern around the world resulting in global debate about how to reduce cement production. Cement manufacturing as a single industry, is estimated to contribute about 6 - 7% of global CO_2 emissions (Aprianti, 2017; Zhang *et al.*, 2017).

Therefore, OPC makes a substantial contribution to global warming and GHGs emissions; finding alternative materials to reduce OPC production has become a vital issue for current and future generations.

Waste, or by-product materials (BMs), are produced in huge quantities every day worldwide. They also have a negative impact on sustainability and the environment due to the cost of disposal and potential contamination to land and groundwater if heavy metals are present as part of their chemical composition (Karim *et al.*, 2013). However, some BMs have high to moderate calcium contents such as sewage sludge ash (SSA), ground granulated blast furnace slag (GGBS) and calcium carbide residue (CCR). Such materials have the potential to play the role of cementitious materials, reacting with silicates through a pozzolanic reaction, resulting in cementitious products similar to those obtained from the OPC hydration process (Sun *et al.*, 2015). Other BMs are rich in silica (Si) and alumina (Al), such as pulverised fuel ash (PFA), silica fume (SF), palm oil fuel ash (POFA) and rice husk ash (RHA) which react pozzolanically with the hydrated calcium compounds (Aprianti, 2017). Numerous research projects have been conducted to utilise BMs to replace a portion of cement in binders and then used in diverse construction projects such as concrete for buildings, rigid pavements and soil stabilisation (Kumar *et al.*, 2007; Jaturapitakkul *et al.*, 2011; Horpibulsuk *et al.*, 2012; Kotwica *et al.*, 2017). Recently, researchers attempted to develop new cementitious binders produced completely from OPC-free blended materials using high calcium waste fly ashes mixed with different types of pozzolanic and alkaline wastes and fly ashes (Sadique *et al.*, 2013; Al-Hdabi *et al.*, 2014; Dulaimi *et al.*, 2017).

In terms of soil mechanics, soft soils are considered problematic because of their undesirable properties associated with low compressive strength, high compressibility and dramatic volume change when their water content changes (Kolias *et al.*, 2005). Soil improvements, specifically soil stabilisation, is the most acceptable technique to mitigate these properties and meet the requirements of engineering projects. Soft soil stabilisation has traditionally been achieved by mixing soft soils with binder materials such as lime, cement and/or fly ash. Studies involving lime and OPC as preferred binder materials, rely on their ability to bind soil particles to each other, resulting in an improved material (Farouk and Shahien, 2013; Jafer *et al.*, 2017).

Substantial quantities of POFA and RHA are produced, worldwide, every year (Aprianti *et al.*, 2015). The disposal and transportation of the solid waste generated from POFA and RHA activity is a serious problem, both environmentally and financially, making it necessary to address this problem with some urgency.

POFA is a pozzolanic waste material from the palm oil industry. It is generated in huge quantities, mainly in developing countries (Karim et al., 2013). Indonesia and Malaysia are the primary POFA producers, manufacturing 86% of global supplies (Aprianti, 2017). Shafigh et al. (2014) reported that Malaysia's annual production of crude palm oil is 7 million tonnes, while a hundred thousand tonnes of POFA are produced by Thailand annually, as reported by Jaturapitakkul *et al.* (2007).

The influence of POFA as a cement replacement, on the compressive strength of mortar, was investigated by Jaturapitakkul *et al.* (2011). POFA was found to be able to enhance the compressive strength of mortars when replacing OPC type I by 10% - 40% by the binder mass. The results showed that because of the efficacy of POFA particles at filling pore voids and its pozzolanic reaction, the compressive strength of mortars increased with an increase in the cement replacement.

In the field of soft soil stabilisation, POFA has been used as a cement replacement in order to improve the Atterberg limits and unconfined compressive strength (UCS) (Pourakbar *et al.*, 2015). Ground POFA was used in two different particle sizes (30µm and 12µm) by Mujah *et* *al.* (2015) for soil improvement. Shear and one dimensional consolidation tests were conducted to evaluate the effect of different sizes of POFA particles in soil stabilisation, the results indicating that fine grade POFA gave a much more pronounced improvement in comparison to the coarse grade. In addition, the internal friction angle and cohesion of the soil when reinforced with both grades of POFA, increased between 50% - 60%. However, there are few, if any, investigations of POFA as a potential pozzolanic activator for calcium based materials for use in soft soil stabilisation.

RHA is a waste that is produced from the incineration processes of rice husk for power generation purposes and rice processing mills (Karim *et al.*, 2013; Mujah *et al.*, 2015). It is produced in huge quantities in the major rice supplier countries such as China, India, Malaysia, Indonesia and Bangladesh. It was reported that approximately 742 million metric tonnes of rice paddies are produced annually by the rice husk harvest by the end of 2013 , while the global annual production of RHA is estimated to be about 7500 thousand tonnes, with an approximate annual growth of 1.1% (Aprianti et al., 2015). RHA is a super-pozzolanic material due to its high silica content (85% to 90%), meaning it can be used as a SCM to produce high performance concrete and geopolymer cement as established by recent research (Hwang and Huynh, 2015; Alex *et al.*, 2016; Sua-iam *et al.*, 2016). Research conducted by Nimwinya *et al.* (2016) used calcined water treatment sludge (WTS) and RHA, activated by alkali solutions of sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3), to produce a sustainable geopolymer binder. The results showed that RHA helped increase the ratio of SiO_2/Al_2O_3 which, in turn, significantly increased the compressive strength of the prepared mortars. The optimum SiO_2/Al_2O_3, at approximately 4.9 and 5.9, provided the highest compressive strength at room temperature and 60°C, respectively.

Flue gas desulphurisation (FGD) gypsum is a waste or industrial by-product material, which is generated from the wet-type desulphurisation processes used in coal-fired power plants; its main phase is calcium sulphate dehydrate (Zhang *et al.*, 2016a). Due to its major component being calcium sulphate, FGD has been used as a grinding agent, instead of gypsum, and to achieve higher early strength by different researchers. Qiao et al. (2006) used FGD mixed with rejected PFA to produce a binder for stabilisation/solidification processes, as the final stages in the treatment of hazardous waste before sending to landfill. The results of strength tests indicated an acceptable development in compressive strength using a binder containing 10% FGD, the strength achieved being suitable for disposal in landfill. A high-calcium waste fly ash, activated by natural alkaline material, was used as a cement replacement. The mortars prepared using this fly ash were found to exhibit higher compressive strengths at all ages of curing, when the FGD was used as 5% of the added binder, as stated by Sadique *et al.* (2013).

In spite of the works mentioned above, there is little if any research using POFA, RHA and FGD to activate a calcium based material in soft soil stabilisation, making this research the first to utilise the aforementioned wastes in a ternary blending system. This research was carried out using 100% replacement of traditional binder (OPC) by waste materials to get benefits both in terms of producing an environmentally friendly binder and to offer substantial economic advantages. The influence of different ternary mixtures produced from mixing HCFA, POFA and RHA at different proportions, on the compressive strength of the stabilised soil, along with compaction parameters and consistency limits, were investigated. The prepared samples of the treated soil were exposed to different curing periods, ranging between 3 and 180 days, to evaluate the short and long term performance of the mixtures. Scanning electron microscopy (SEM) testing and energy dispersive X-Ray spectroscopy (EDX) analyses were carried out on the developed binder paste to analyse the improvement gained in the geotechnical properties of the stabilised soil.

2. Materials and Methods

2.1 Materials

2.1.1 Soil samples

The soil used in this study was collected from the shoulder of the River Alt estuary, located in Hightown to the north of Liverpool, UK. This type of soil covers a very large area in Hightown as well as the coastal area of the river and can be used for a variety of applications in different construction projects. The soil samples were collected from depths ranging between 0.3m to 0.5m below ground level. They were put in well-sealed bags of 20 to 25kg and sent to the soils laboratory. Figs. 1a and b show the satellite image of the site and the corresponding location where the soil samples were extracted.

Once the soil samples arrived at the laboratory, representative samples were taken to determine the natural moisture content. The rest of the soil samples were air dried for approximately four days, followed by 24hrs of oven drying at 110 ± 5^{o}C. After drying, soil lumps were fragmented using a light wooden hammer then passed through a sieve of size 3.35mm. Table 1 illustrates the main physical and engineering properties of the virgin soil (VS) used in this study. The grain size distribution revealed that it was composed of 13.1% sand, 43.9% silt and 43.0% clay, making the VS silty clay. The liquid limit and plasticity index were found to equal 44.0% and 20.2 respectively, while the organic matter content obtained from the loss of ignition test was 7.95%. Based on this analysis, and according to BS EN ISO 14688-2:2004+A2013 (European Committee for Standardization, 2013), the VS used in this study is classified as an intermediate plasticity silty clay with sand (CI).

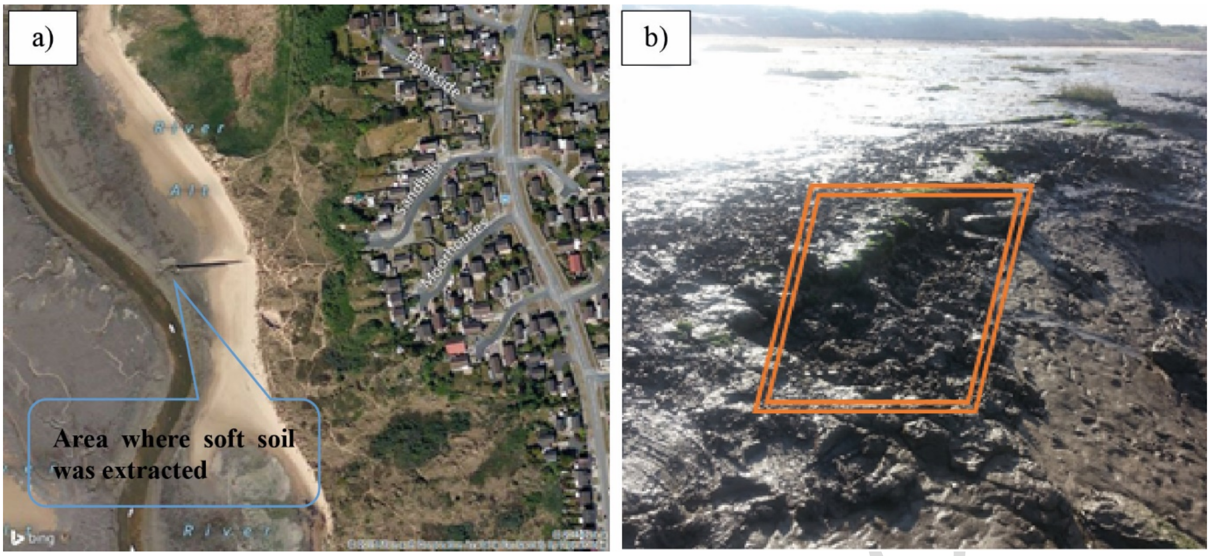

Fig. 1. (a) The satellite image of the site from where the soil was extracted, (b) soil extraction location (location coordinates are 53°31'03.4"N 3°03'48.2"W).

Table 1. Main physical and geotechnical properties of the virgin soil used in this study.

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 %	43.0
Specific Gravity (Gs)	2.57
Maximum dry density (MDD) Mg/m^3	1.57
Optimum moisture content OMC %	23.0
pH	7.78
Organic Matter Content %	7.95
Unconfined Compressive Strength (UCS) kPa	66.5

Mg/m^3= Mega gram/cubic metre, kPa = kilopascal.

2.1.2 Binder Materials

The materials used to produce the binders in this study were high calcium fly ash (HCFA), palm oil fuel ash (POFA), rice husk ash (RHA) and flue gas desulphurisation (FGD) gypsum. HCFA is obtained from power generation plants using an incineration process at temperatures between 850°C and 1100°C by means of a fluidised bed combustion system (FBCS). The POFA was imported from the Sg. Tengi Palm Oil Factory, Kuala Kubu Bharu, Selangor, Malaysia, as a waste material produced from the incineration processes applied to palm oil fibres at temperatures ranging between 800°C and 1000°C. It was treated by passing through a sieve size of 150μm to remove the incomplete incinerated materials then ground for 15 minutes using a pestle and mortar grinder to increase the pozzolanic reactivity. A similar procedure was applied on POFA prior to being used in the experimental work by Awal and Shehu (2013). The RHA was supplied by NK Enterprises Company, Jharsuguda, Orissa, India, while the FGD gypsum was provided by the soils laboratory in LJMU. The reference cement used in this study was a commercially available cement type CEM-II/A/LL 32.5-N supplied by Cemex Quality Department, Warwickshire, UK. The chemical compositions of the binder materials utilised in this study are listed in Table 2. The oxide contents were obtained by conducting the X-Ray florescence spectrometry (XRF) analysis using a Shimadzu's EDX-720 Energy Dispersive X-Ray Fluorescence Spectrometer.

Table 2 shows the substantial calcium oxide content of HCFA which, with the silica oxide content, is comparable to those for the reference cement. The chemical compositions of the HCFA in this study are consistent with those of Dulaimi *et al.* (2016) but the CaO content of the HCFA (66.76%) is higher than that reported by Sadique *et al.* (2012a). Based on the chemical compound analysis, HCFA has a promising enough CaO content to have the potential to form the basis for the cementitious material as it can react with pozzolanic materials to produce cementitious compounds. The main oxides of POFA are in line with those reported by Chindaprasirt *et al.* (2014) with slight differences in K_2O and Al_2O_3 content. With respect to the RHA, SiO_2 is the main oxide (90%+), this the same as the chemical composition reported by Karim *et al.* (2013); however, there is more SiO_2 in the current research sample. The pozzolanic materials in POFA and RHA, represented by SiO_2 and Al_2O_3, promise to boost the pozzolanic reaction during the hydration process. It has been reported that silicates and aluminates react chemically with the hydrated lime $Ca(OH)_2$ in cement in the presence of moisture, forming more cementitious gel such as calcium silicate hydrate (C-S-H) and calcium aluminate hydrate (C-A-H) (Jiang *et al.*, 2016). The chemical compositions in Table 2 show that the FGD was composed mainly of CaO and sulphate (SO_3) with a small amount of SiO_2. A similar chemical composition for FGD was reported by Zhang *et al.* (2016b).

Table 2. Chemical properties of the materials used in this study

Material	pH	LOI %	Chemical composition %							
			CaO	SiO_2	Al_2O_3	Fe_2O_3	MgO	Na_2O	K_2O	SO_3
HCFA	12.86	N/A	66.76	25.12	2.38	0.03	2.57	1.718	0.31	0.38
POFA	13.04	2.78	9.047	53.01	6.487	4.873	1.329	1.495	6.501	2.531
RHA	8.98	2.05	0.493	90.20	4.03	0.183	0.609	0.90	1.36	-
FGD	12.3	-	35.89	14.3	-	-	0.54	1.23	-	34.64
OPC	13.04	0.28	66.12	24.91	1.728	1.668	1.322	1.357	0.836	2.655

2.2 Experiments and testing program

2.2.1 Compaction parameters test

The standard Procter compaction test was performed to determine the maximum dry density (MDD) and optimum moisture content (OMC) for the virgin soil and the soil treated with different binders. The procedure followed is fully explained in BS 1377-4:1990 (British Standard, 2002). Sample pastes made from the binder-soil mixtures, were mixed and prepared after adding water, then compacted using an electrical compactor. This test is essential to prepare the samples required for other geotechnical experiments such as the unconfined compressive strength (UCS) test.

2.2.2 Atterberg limits

This test was carried out according to BS 1377-2:1990 (British Standard, 1998) to determine the liquid limit (LL), plastic limit (PL) and plasticity index (PI) of the untreated and treated soil with different binders. Samples of the treated soil were prepared by adding a specified quantity of binder, then mixed manually with soil for not less than 5 minutes or until a homogenous colour was apparent. The test was performed immediately after adding water to the binder-soil mixtures. The cone penetration method was used to determine the LL using a cone penetrometer devise.

2.2.3 Unconfined compressive strength test

The unconfined compressive strength (UCS) test is one of the most common tests used to evaluate the strength of the soil and its suitability for use in different civil engineering projects. This test was performed using a computerised and motorised triaxial machine but without applying any lateral load in the triaxial cell ($\sigma_3 = 0$). The test procedure described in BS 1377-7:1990 (British Standard, 1999) was adopted. Specimens of the binder-soil mixtures, with specific dimensions of diameter 38mm and height 76mm, were prepared using a fixed volume mould. Three specimens were prepared for each corresponding dose of additives by compressing the soil-binder mixture inside the fixed volume mould using a manual hydraulic compression rig. Following this, the specimens were removed from the mould, weighed, wrapped in cling film, labelled, placed in well-sealed plastic bags and stored for curing at room temperature (20 ± 2^{o}C).

2.2.4 Testing program and mixing proportions

The first stage of the experiments comprised the optimisation of the binder content using the HCFA alone as a binder. This stage was dependant on the results of the UCS tests which were conducted on specimens of soil treated with different percentages of HCFA (0, 3, 6, 9, 12 and

15% by the dry mass). The specimens were kept for different curing periods (7, 14 and 28 days) prior to UCS testing. The second stage was for both ternary blending optimisation and comparison of the binders' performance with that of the soil treated with the reference cement (OPC). The performance of different binders was evaluated dependant on the results of the Atterberg limits, compaction parameters and UCS tests. The comparative UCS testing was carried out on the specimens at 3, 7, 28, 90 and 180 days. Table 3 illustrates the mixing proportions adopted to produce different binder mixtures from the waste materials used in this study. The mixtures containing FGD were ground at 15 minutes using a pestle and mortar grinder with low agitation (1 horsepower) and a bowl capacity of 2.5 litres.

Table 3. Mixing proportions used to produce different blended binders.

Binder ID	OPC %	HCFA %	POFA %	RHA %	FGD %
VS	-	-	-	-	-
U	-	100	-	-	-
BB1	-	75	25	-	-
BB2	-	75	-	25	-
TB1	-	75	12.5	12.5	-
TB1-FG	-	75	12.5	12.5	5
TB2	-	66	17	17	-
TB2-FG	-	66	17	17	5
RF	100	-	-	-	-

RF is for reference, U is for unary mixture which contains HCFA only, BB and TB are for the binary and ternary blended binders respectively, and FG is for the binders treated with FGD

2.3 Techniques for the analytical characterisation

2.3.1 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}. This analysis was conducted on the raw materials, in their dry powder state, and on the hydrated pastes of the most remarkable binder mixture (TBCB) along with the reference cement. The hydrated pastes were subjected to 3, 28 and 180 days of curing prior to XRD analysis. The diffraction patterns of the undisturbed raw materials shown in Fig. 2 indicate that the mineralogy of HCFA was crystalline without significant background noise (Fig. 2a); the major crystal peaks were calcite ($CaCO_3$), lime (CaO), gehlenite ($CaAl[AlSiO_7]$), merwinite ($Ca_3Mg[SiO_4]$), mayenite ($Ca_{12}Al_{14}O_{33}$) and quartz (SiO_2) (Fig. 2a). The dominant mineral phase of POFA was quartz (SiO_2), while cristobalite (SiO_2) and potassium aluminium phosphate ($K_3Al_2[PO_4]_3$) were identified as minor phases, similar to the diffraction patterns of the POFA as reported by Karim *et al.* (2013) (Fig. 2b). The XRD analysis of RHA shown in Fig. 2c reveals an amorphous nature which is expected to show high reactivity during the hydration reaction. Finally, a crystalline nature was observed for the FGD gypsum with calcium sulphate hemihydrate ($CaSO_4.0.5H_2O$) as the dominant mineralogical phase (Fig. 2d).

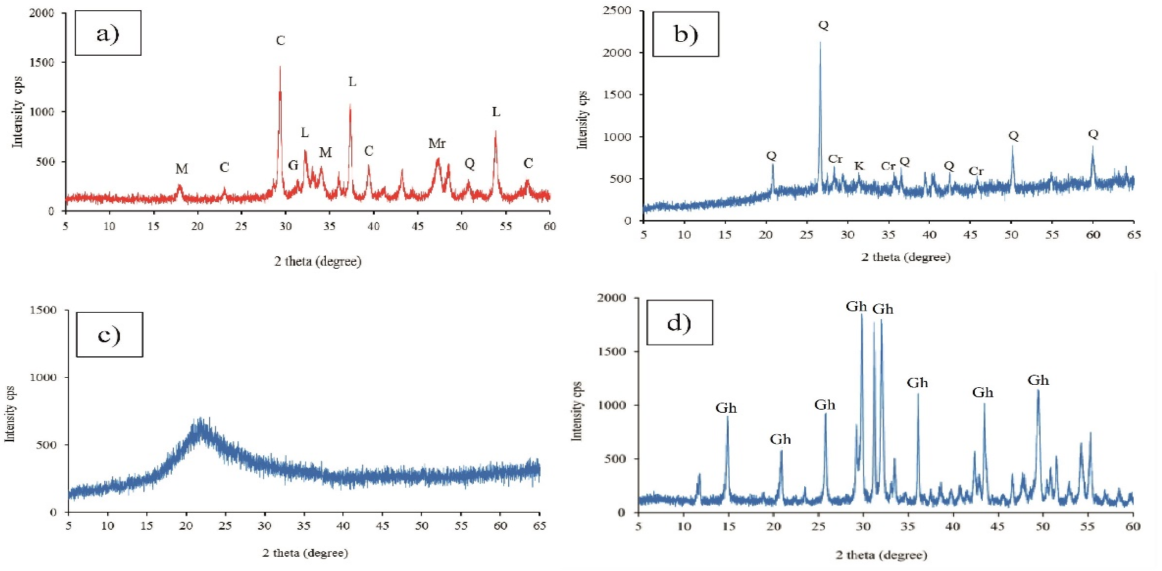

Fig. 2. Diffraction patterns of the waste materials used in this research; (a) HCFA, (b) POFA, (c) RHA and (d) FGD gypsum. C: calcite, L: lime, G: gehlenite, M: mayenite, Mr: merwinite, Q: quartz, Cr: cristobalite, K: potassium aluminium phosphate and Gh: calcium sulphate hemihydrate.

2.3.2 Scanning electron microscopy (SEM) observation

The SEM imaging technique was carried out on the raw materials in their powder states and on the hydrated pastes of TBCB and RF. This test was undertaken using an EDX Oxford Inca x-act detector, an FEI SEM model Inspect S and a Quanta 200 with an accelerating voltage of 5-20kV. Prior to SEM imaging, the specimens were coated with a thin layer of Palladium using a sputter coater for increased visibility. The samples of the hydrated pastes were allowed 3, 28 and 180 days of curing before performing the SEM imaging.

Fig. 3 shows the microphotographs of the raw materials used in this study. The HCFA particles are agglomerated and have a coagulated state occurring in clusters. 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. POFA has spherical-shaped particles with some irregular-shaped particles with sharp angles while the SEM observation of RHA revealed that its particles tend to have an irregular shape with sharp angles. FGD gypsum particles were found mainly to be large tabular-shaped with a small number of small irregular-shaped particles. The large size particles are gypsum as reported by Zhong *et al.* (2012)

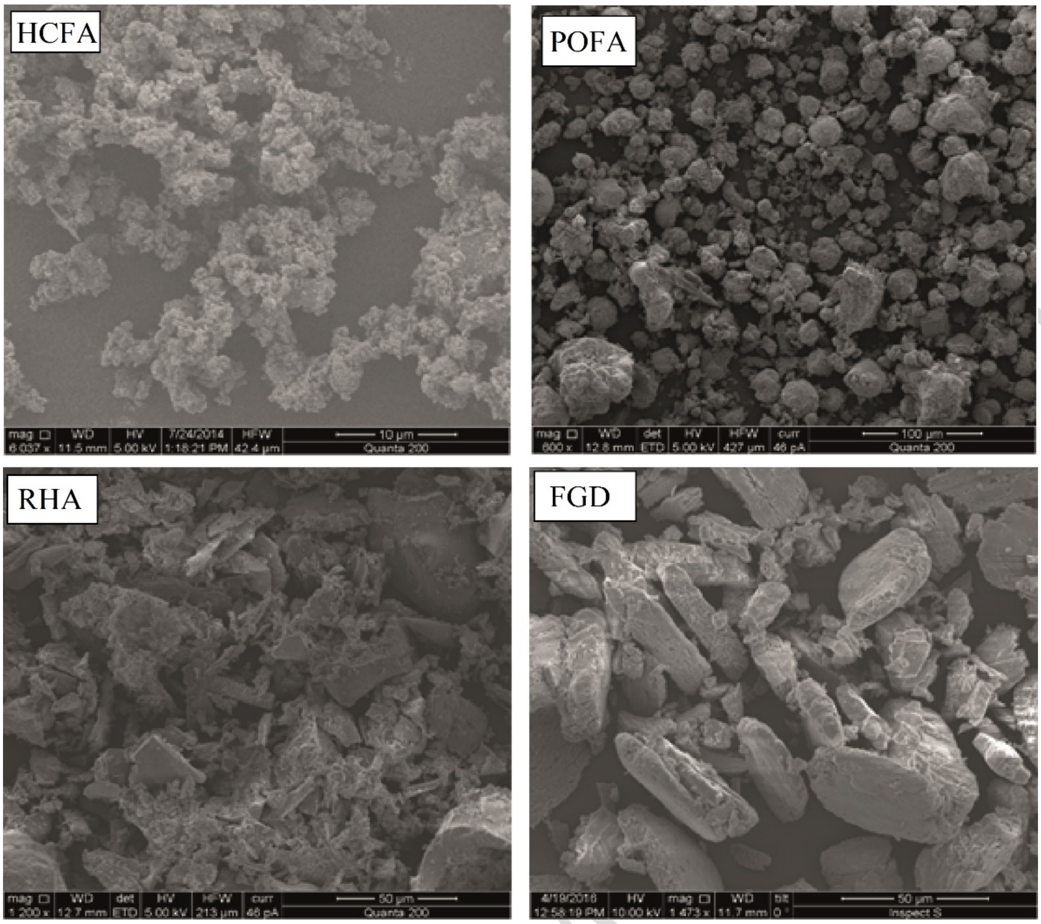

Fig. 3. SEM images of the raw materials

3. Results and discussion

3.1 Optimisation of the binder content

The optimisation of the binder content was carried out using HCFA alone as an additive to the soft soil, dependant on the results of the UCS tests. The compaction tests were conducted prior to the UCS tests, to determine the MDD and OMC for each dosage of HCFA used to prepare the corresponding specimens for the UCS tests. Fig. 4 illustrates the effect of the HCFA content on the MDD and OMC of the treated soil; the MDD decreased and OMC increased significantly with the increase in HCFA content. The MDD for the virgin soil decreased substantially from 1.56Mg/m^3 to 1.40Mg/m^3 when adding 15% of HCFA by dry mass. This percentage increased the OMC significantly from 23% for the untreated soil to 30.5%. This behaviour may be attributed to the substantial CaO content of HCFA which increases the water demand of the soil-binder mixture, this high water demand leading to a higher OMC value (Jauberthie *et al.*, 2010). The cation exchange which occurs immediately after adding water between the clay minerals of the treated soil and CaO's positive ions, may also contribute to the increase in water demand of soil-binder mixtures (Eskisar, 2015).

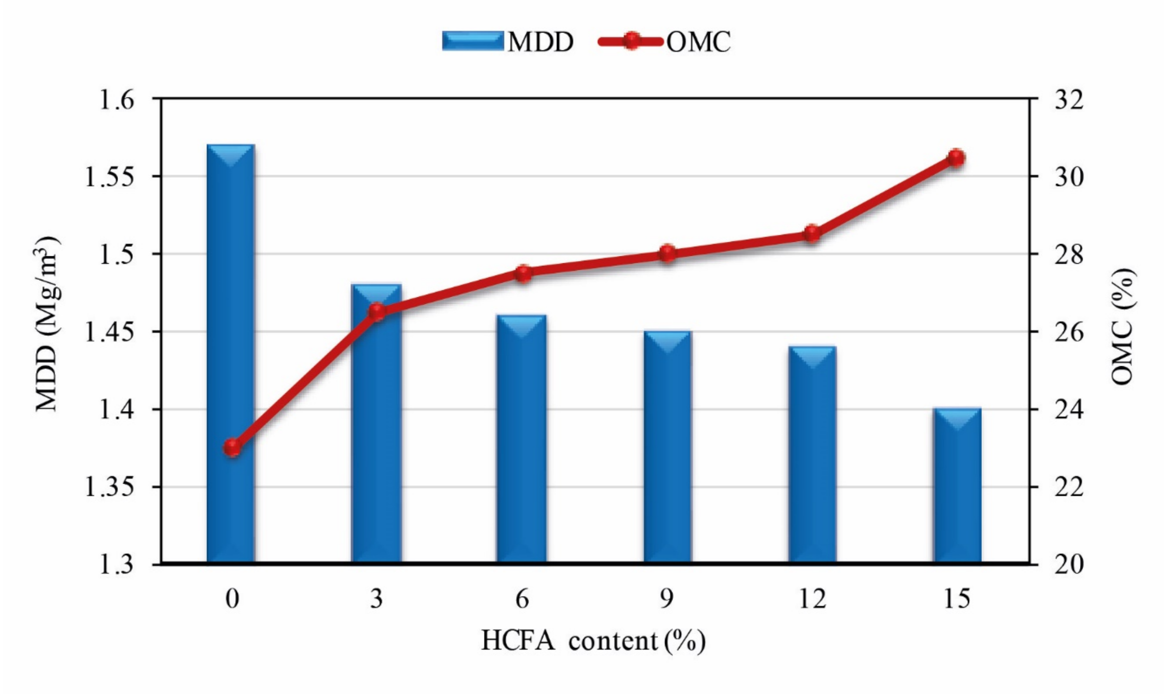

Fig. 4. Effect of HCFA content on MDD and OMC

Fig. 5 shows the results of the UCS tests on soil treated with different portions of HCFA and cured over various times. Significant increments in the soil compressive strength were achieved with the use of HCFA. The UCS was found to increase with a continuous increase in HCFA content and curing time. However, a reduction was observed in UCS with the use of 15% HCFA when compared with that for the soil treated with 12%, indicating that the optimum binder contented is 12% by dry mass of the treated soil as this facilitates a higher compressive strength across all curing times. At 28 days of curing, the UCS increased from 200kPa for the untreated soil, to almost 700kPa with the use 12% of HCFA.

The flocculation phenomenon, in addition to the cation exchange that occurred between clay mineral cations in the stabilised soil and the divalent calcium ions of the HCFA, may have been responsible for the early strength improvement (James and Pandian, 2016). With the increase in curing time, the development in the soil's compressive strength can be attributed to the pozzolanic reaction occurring between the hydrated lime in HCFA and the silicates and aluminates of the treated soil, resulting in cementitious products that produced a coherent and compact structure (Aïtcin, 2016; James and Pandian, 2016). These products include calcium silicate hydrates (C-S-H) and calcium aluminate hydrates (C-A-H) which are a result of the pozzolanic reaction.

The optimum content (12% HCFA) obtained from this stage of the experiment, was used as the total binder dose to be added to the stabilised soil for the later ternary blending optimisation processes. The same percentage was also used for the reference binder (OPC).

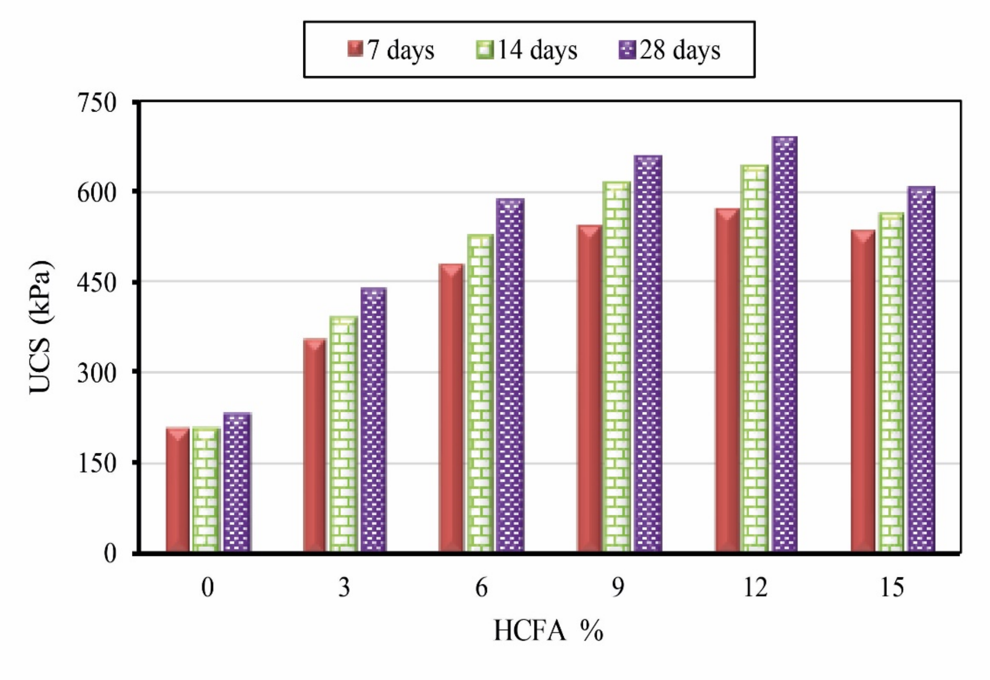

Fig. 5. UCS of the soil treated with different proportions of HCFA at different curing times.

3.2 Ternary blending optimisation and developing of TBCB

3.2.1 Atterberg limits

The results of the Atterberg limits test shown in Fig. 6, indicate that almost all liquid limits (LLs) and plastic limits (PLs) increased significantly for the treated soil in comparison to the virgin soil. Plasticity indices (PIs) decreased with the treatment because of the greater increment in PL, with respect to LL, for each different proportion of additive. The highest value of LL was obtained from the soil treated with U (100% HCFA) due to the high CaO content, slight reductions occurring after adding the blended mixtures. LL values are related to the HCFA content in the binder; a higher HCFA content, gave higher LL and PL. Accordingly, the PI decreased in relation to the difference between LL and PL for the corresponding binder type, this promoting an increase in soil workability, as indicated by Baran *et al.* (2001). The results of the Atterberg limits tests indicated that while the lowest value of PI was obtained from TB2-FG (11.74), all mixtures recorded PIs lower than that for the RF (14.5).

Interestingly, the addition of FGD gypsum as a grinding aid (GA) and sulphate activator to the ternary blended binders (TB1 and TB2), contributed to further reductions in the plasticity index. There were slight reductions in both LL and PL of the soil treated with ternary mixtures aided by FGD, leading to a further reduction in PI. The reduction in LL can be attributed to the decrease in water demand of the binder after using FGD in parallel with the grinding technique. The use of grinding with FGD as a GA, boosts the speed at which ground particles of the activated fly ash dissolve when mixed with water. In turn, water demand is decreased, this increasing the reactivity of the activated fly ash (Sadique *et al.*, 2012a). FGD also assisted in breaking up large plerospheres (i.e. a reduction in porous particles) which in turn, decreased particles roughness. This is of benefit for the grinding process, decreasing water demand as argued by Aydın *et al.* (2010). This reduction in PI increases the soil-binder mixture workability as well as increasing the soils' resistance to swelling and shrinkage stresses.

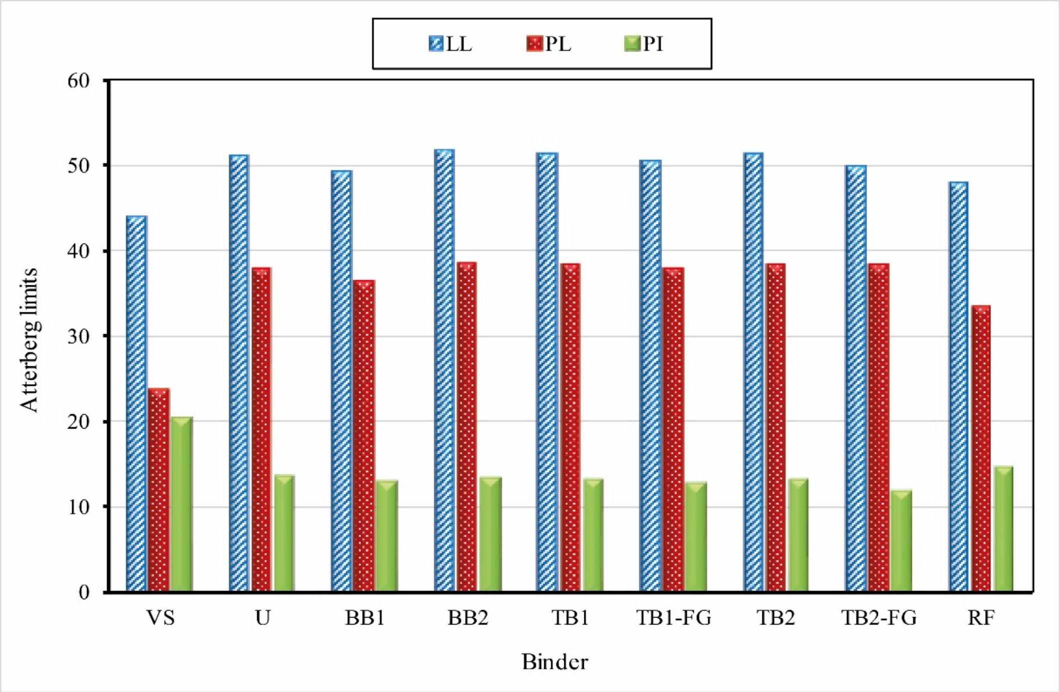

Fig. 6. Atterberg limits for the soil treated with different types of binder

3.2.2 Compaction parameters

Fig. 7 shows the MDD and OMC values for the untreated soil and soil the treated with different binders, obtained from the standard Proctor compaction test. Overall, the MDD decreased and the OMC increased for all soil-binder mixtures in comparison to the virgin soil, this due to the presence of CaO in the binder which leads to a rise in water demand. A substantial reduction in MDD, with a significant increase in OMC, was observed for the U mixture (100% HCFA). There were gradual increments in MDD, accompanied by continuous reductions in OMC, for all the blended mixtures except the soil treated with BB2 which recorded the lowest MDD and highest OMC. This behaviour was due to the proportion of RHA (25%) in this binder, which may be due to the morphology structure of RHA, a high presence of porous and spongy particles and a high water demand (Karim *et al.*, 2013). Although the MDDs increased after treatment with the blended binders, the highest value being achieved by TB2-FG (1.49Mg/m^3), they are still much lower than that of the soil treated with OPC (RF mixture) which was 1.53Mg/m^3. The OMC values obtained from the compaction test were related to the corresponding values of LL and PL, presented in the previous section, in which the binders with high LL and PL also had a high OMC and vice versa. A similar scenario was reported by Garzón *et al.* (2016) when studying the effect of lime on phyllite clay stabilisation. The reduced OMC in the case of mechanically activated ternary blended binders were attributed to particle packing by fine ground particles that act as a filler for the void spaces which also reduced inter-particle friction (Malhotra and Mehta, 2012). Similar improvement relating to reduced water demand after mechanical activation of HCFA was reported by Sadique *et al.* (2013).

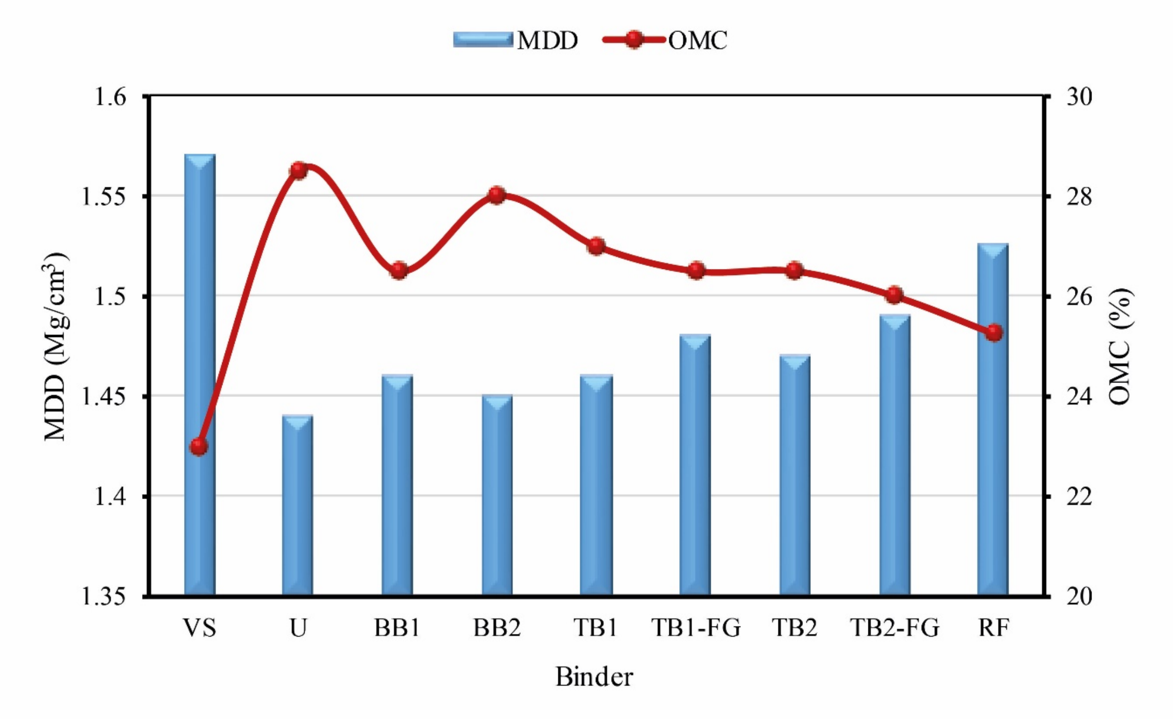

Fig. 7. MDD and OMC of the soil treated with different types of binders

3.2.3 Unconfined compressive strength (UCS)

The comparative UCS values of the stabilised soil are presented in Fig. 8. It was evident that the compressive strength increased noticeably over 180 days of curing for all types of mixtures tested. However, the degree of strength development varied significantly among binder types. In general, the soil specimens treated with blended binders had compressive strengths higher than the soil treated with U at all ages. However, all mixtures showed compressive strengths lower than that for the soil treated with RF for curing periods up to 90 days. At 180 days of curing, a very impressive compressive strength was obtained for the soil samples treated with the TB2-FG mixture (1464kPa) which exceeded that for RF (1450kPa). Even after a short period of curing (3 days), an increase in compressive strength was evident for all types of mixtures. Unlike the other binders, TB2-FG had a UCS of 923kPa at 3 days of curing which exceeded all the other UCS values for soil samples treated with the other mixtures, except RF, even after 28 days of curing.

The increase in compressive strength achieved after the treatment with the binary mixture BB1 (75% HCFA + 25% POFA) was due to an increase in cementitious products because of the superior pozzolanic reaction between POFA silicates with the hydrated lime produced from the HCFA (Aïtcin, 2016). The pozzolanic materials in POFA, along with the high pH value, facilitated the high UCS by boosting the chemical activation of HCFA, resulting in the formation of extra cementitious products (C-S-H). Pourakbar *et al.* (2015) reported a similar improvement in the UCS of soil by using 10% binder of a binary mixture containing cement 80:20 POFA. The soil treated with BB2, which contained 25% RHA in the total binder, showed compressive strengths slightly lower than those obtained from the soil treated with BB1 (Fig. 7). This may be due to the density of the samples treated with BB2 as it was lower than those treated with BB1. Nicholson (2015) reported that an increase in the MDD of treated soil might contribute to an increase in the compressive strength of the soil and improvements in other geotechnical properties. The pure silica provided by RHA in ternary mixtures was found to be beneficial for strength development. The latter has a susceptibility to dissolve and react chemically with hydrated lime, resulting in an increase in cementitious products formation as shown in Fig. 8 for TB1 and TB2.

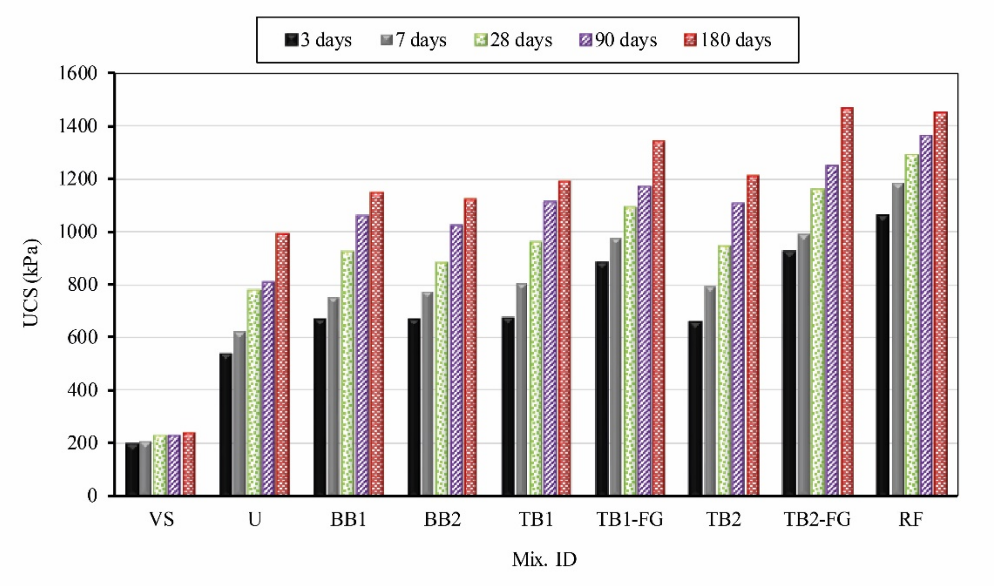

Fig. 8. Unconfined compressive strength of the soil treated with different binders at different curing times

The high alkalinity of POFA and an increase in the pozzolanic materials content in the total binder, as in the mixture TB2, ensures the generation of sufficient pozzolanic materials which in turn boosts the pozzolanic activation of the hydrated lime from HCFA. Puppala *et al.* (2015) reported that a high alkaline environment for the chemical reaction of binders is preferable as this boosts hydration and pozzolanic reactions. Such circumstances accelerate the solution of the glassy phases of silicates and aluminates and provides extra silica that has the capacity to react with hydrated lime resulting in more cementitious compounds. Similar findings were reported by Al-Hdabi *et al.* (2014) when they developed a new cold Bitumen Emulsion mixture (CBEM) using supplementary cementitious materials to replace the lime (the conventional filler) in such mixtures.

With respect to the results achieved after the application of grinding using FGD as a GA, FGD helped increase the compressive strength significantly after relatively long curing periods (Sadique *et al.*, 2013; Zhang *et al.*, 2016b). The evolution achieved in UCS after using FGD can be attributed to the dual role of FGD gypsum by increasing the reactivity of fly ash particles during the inter-grinding due to the increment in the specific surface area and by sulphate activation by providing SO_4^{2-} during the hydration reaction. FGD depolymerised the glass structure of fly ash facilitating additional penetration of Ca^{2+} and SO_4^{2-} resulting in an accelerated pH and boosted dissociation of the fly ashes glass phase. This led to enhance the hydration and pozzolanic reactions resulting in the production of more cementitious products, hence, the compressive strength increased (Sadique *et al.*, 2012b; Wright and Khatib, 2016). A similar finding has been reported by Zhang *et al.* (2016b) regarding a composite cementitious system produced from FGD gypsum combined with metakaolin-cement, where the compressive strength of the mortars of this composite exceeded that for the mortars prepared using pure cement, after 120 days of curing.

In summary, the TB2-FG mixture is the one chosen as the TBCB to be used for subsequent XRD analysis and microstructural investigation.

3.3 Comparative XRD analysis and SEM observation of the hydrated pastes of TBCB and RF

3.3.1 XRD analysis

A chemical synergy was apparent in the mechanically activated ternary blend (TBCB), where pozzolanic and hydraulic properties between the blended materials were compensated and balanced by each other (Fig.9 – dry powder). Gartner and Macphee (2011) also reported that a combination of oxide phases was necessary, that is metastable in presence of water and tends to convert to a more stable phase assemblage when hydrated. The presence of aluminium and potassium ions, supplied by POFA, and amorphous silica and aluminium ions supplied by RHA, were expected to accelerate the dissolution of HCFA by reducing the concentration of Ca^{+2} and Al^{+3} in the mix to form ettringite and alunite and hence solidify the system.

In terms of the hydrated states, the phase composition of the TBCB and control cement hydration products were identified in the XRD analysis as shown in Fig. 9 and Fig. 10 respectively. In the case of TBCB, the hydrates were identified as C-S-H, portlandite

[$Ca(OH)_2$], ettringite [($Ca_6Al_2(SO_4)3(OH)_{12}\cdot 26H_2O$], alunite [($KAl_3(SO_4)2(OH)_6$], which were found to intensify with age. This indicates accelerated hydration and subsequent progressive strength development. Moreover, as for the reference cement (Fig. 10), the rapid consumption of mineral phases with reduction of crystallinity, the appearance of new crystal peaks of hydrates, and conversion of the silicate phase to CH phase was in accordance with the observation stated by Esteves (2011) and Esteves (2011) concerning the XRD study of cementitious material upon hydration. The main strength generating reaction product of cementitious materials is C-S-H. In the case of TB2+FG paste, the characteristic peak corresponding to C-S-H has been termed as C-Al/K-S-H in the XRD diffractogram as shown in Fig. 9. Similar combinations of calcium sodium/potassium silicate hydrate were reported by Guo *et al.* (2009) and Sadique *et al.* (2013).

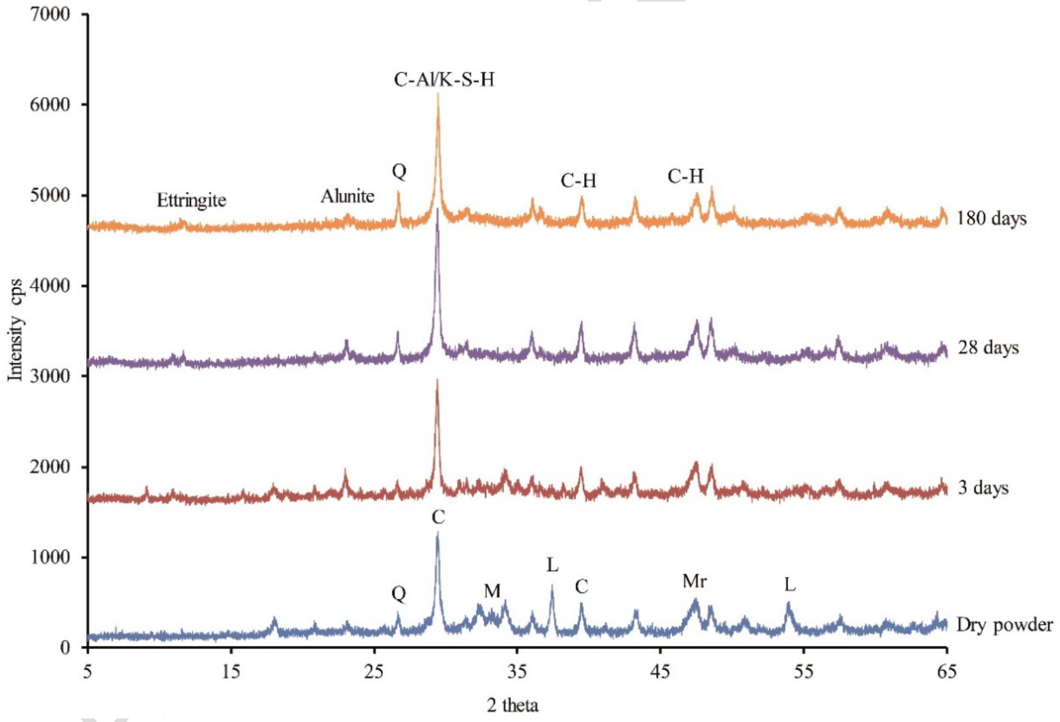

Fig. 9. XRD patterns of the hydrated pastes of TBCB at different curing periods. C: calcite, L: lime, M: mayenite, Mr: merwinite and Q: quartz

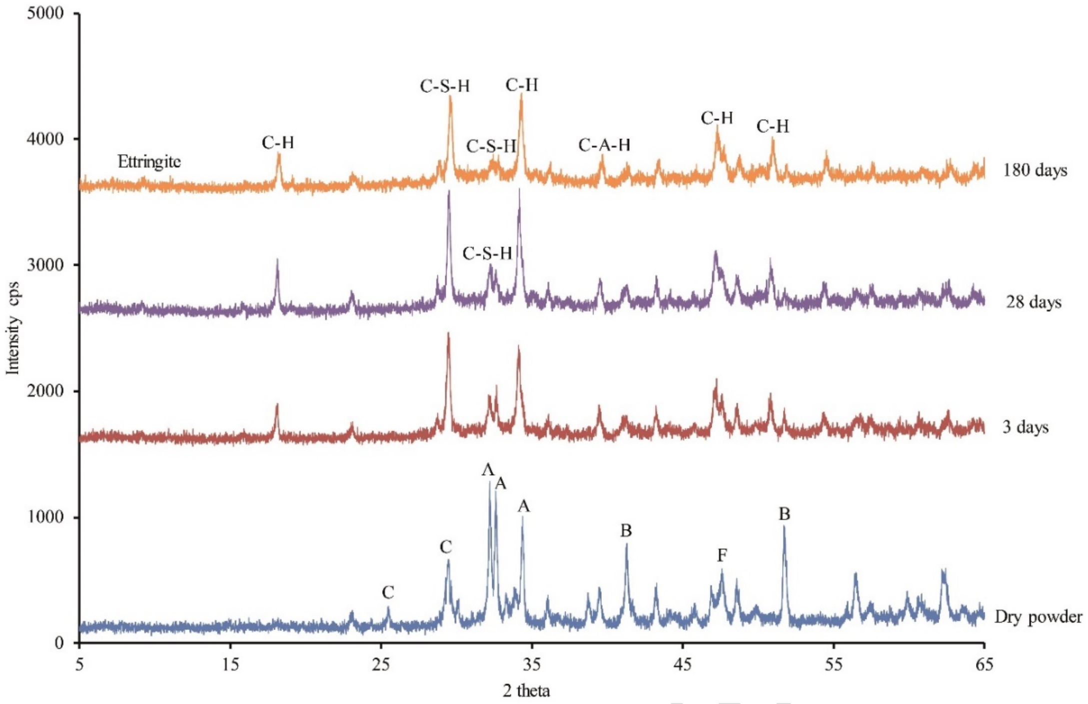

Fig. 10. XRD patterns of the hydrated pastes of RF at different curing periods. C: calcite, A: alite, B: belite, and F: ferrite

3.3.2 SEM observation

Fig. 11 and Fig. 12 show the micrographs of the TBCB and RF paste samples after 3, 28 and 180 days of curing respectively. In the case of TBCB, the formation of hydration products, specifically the flaky shaped crystals (Portlandite (CH)), was clearly evidenced at 3 days of curing. SEM analysis revealed a more pronounced microstructure after 3 days of curing; the hydration products CH, C-S-H gel and needle-shaped particles (ettringite) were clearly observed at 28 and 180 days of curing. No intact powder particles of raw materials were detected; the HCFA, POFA and RHA particles were transformed into hydration products due to a successful hydration reaction (Fig. 11). A compacted and coherent structure was achieved which was consistent with the development gained in the compressive strength of the stabilised soil as well as with the results obtained from the XRD analysis for TBCB. The consequent changes in the microstructure of the TBCB paste was comparable to that for the reference cement shown in Fig. 12. However, the SEM images indicated a denser microstructure for the cement paste with less CH crystals detected for the curing periods up to 28 days. After 28 days of curing, most of the ettringite and CH were found to have been consumed due to the successful pozzolanic reaction, the surface of the TBCB paste almost completely covered by C-S-H gel. A very dense microstructure of TBCB was observed at 180 days of curing, which was similar to that of RF, where the hydration products, specifically C-S-H gel, were found mainly in the pore areas of the sample. The greater profusion of CH crystals and ettringite in RF compared to TBCB, after 180 days of curing, (Fig. 11 & Fig. 12) was found to be consistent with their corresponding XRD analyses (Fig. 9 & Fig. 10).

Moreover, in case of TBCB, the conversion of ettringite to Monosulfoaluminoferrite (AFm) was also detected at 28 days of curing, appearing as lath-like crystals. AFm appears after a period of low activity due to the slow diffusion of species in the hardened materials which experience a chemical reaction between ettringite and C_3A (Marchon and Flatt, 2016). The formation of ettringite, and the more pronounced AFm at 28 days, can be attributed to the existence of FGD gypsum which contains a significant amount of sulphates; similar findings were reported by Jha and Sivapullaiah (2016).

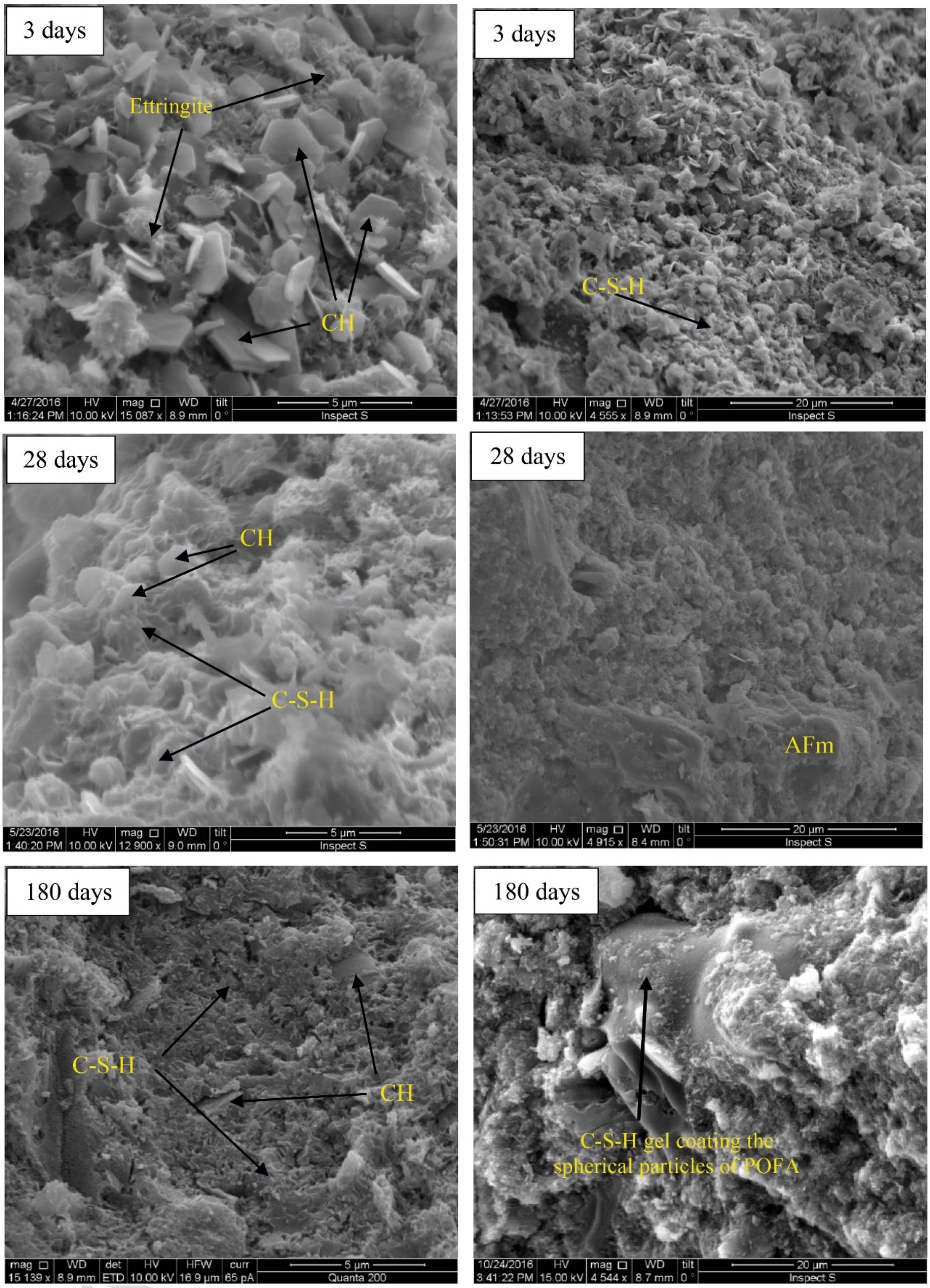

Fig. 11. The micrographs of the TBCB paste at 3, 28 and 180 days of curing.

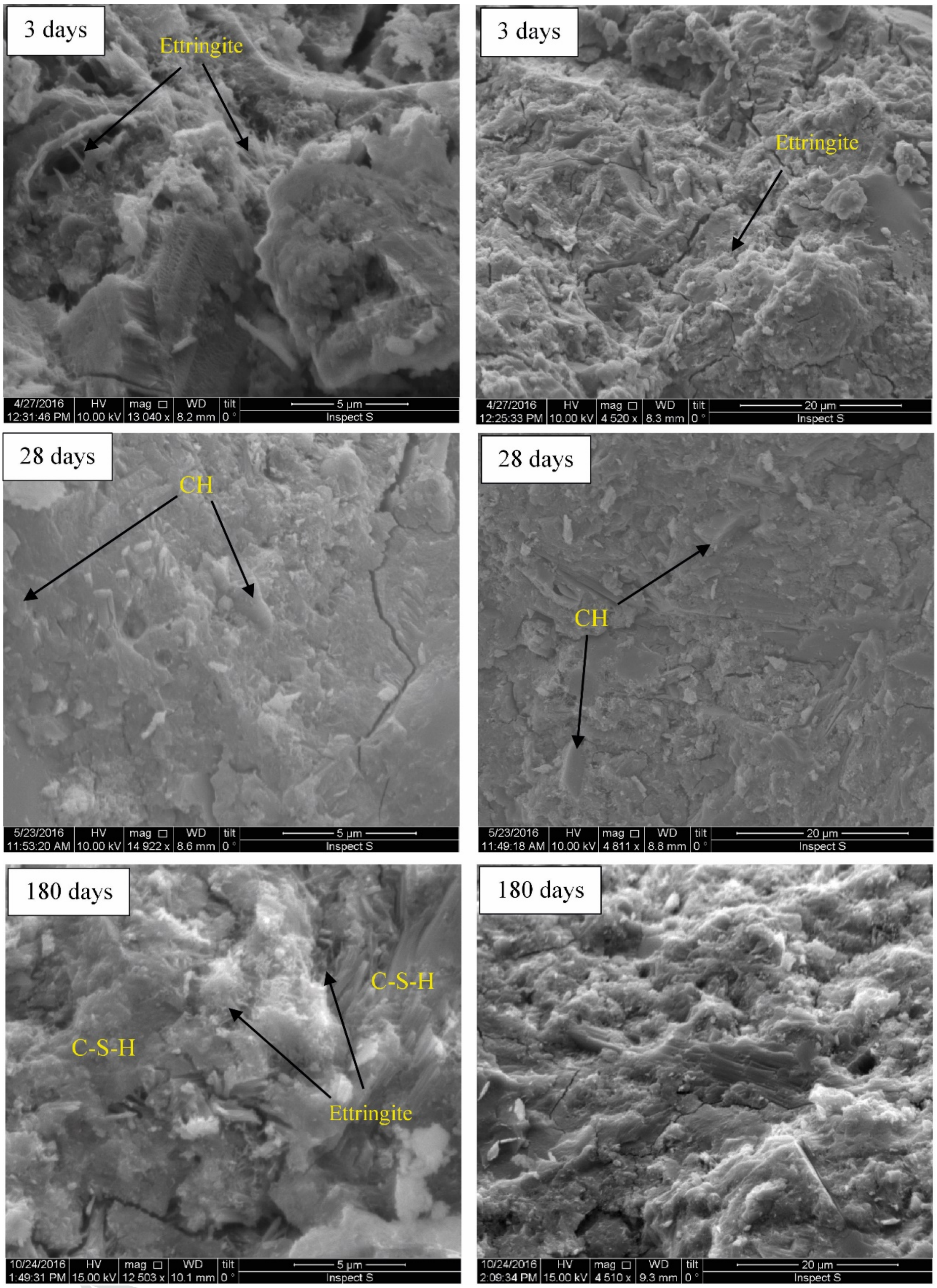

Fig. 12. The micrographs of the RF paste at 3, 28 and 180 days of curing.

4. Conclusions

The effect of high calcium fly ash, along with its pozzolanic activation using POFA and RHA on the mechanical properties of a soft soil, were examined in this study. The use of FGD gypsum, as a grinding agent and retarder to boost the chemical reaction and develop compressive strength, was also investigated. This is a novel approach for the use of waste materials to develop a new, sustainable, ternary blended cementitious binder (TBCB) for use in soft soil stabilisation. Based on the results obtained in this study, the following conclusions can be drawn:

1. A new, ternary blended cementitious binder (TBCB) produced from waste materials was developed from 66% HCFA + 17% POFA + 17% RHA, activated with 5% FGD gypsum, by total binder. This binder can be used for commercial cement replacement in soft soil stabilisation which, in turn, contributes to the reduction of the negative environmental footprint created by the manufacture of cement.
2. In terms of soil consistency limits, significant improvements in Atterberg limits were achieved using TBCB, which were even better than those obtained from the soil treated with the reference cement. By using TBCB, PI decreased significantly from 20.22 for the untreated soil to 11.74 for soil treated with TBCB which was less than that for soil treated with RF (14.5). This will significantly improve soil resistance against swelling and shrinkage stresses.
3. FGD gypsum contributed significantly to improve the soil consistency limits when they are compared with the soil treated with non-mechanically activated binder (TB2). A further reduction in PI was achieved with the FGD activation which recorded the lowest PI value (11.74). Additionally, FGD gypsum was found very beneficial in the development of the

soil compressive strength as it contributed to the acceleration of the dissolution of the silicates which led to enhance the pozolanic reaction.

4. UCS of the soft soil at a short curing period (3 days) noticeably increased with the use of TBCB. The UCS of the stabilised soil increased from 200kPa for the virgin soil to 923kPa by using TBCB; this is 4.6 times the UCS of the untreated soil. This value represents 87.24% of the UCS of the soil treated with reference cement (1058kPa). This improvement can facilitate the provision of a strong enough soil base for labour and equipment at building sites within a very short time.
5. With respect to the compressive strength at prolonged curing periods, substantial developments in UCS were achieved, specifically for the samples treated with TBCB. Their UCS exceeded that for the samples treated with RF at 180 days of curing. A UCS of 1464kPa was recorded for the soil treated with TBCB; the soil treated with RF achieved 1450kPa. This represents a significant improvement in soil strength by 7.32 times that for compacted virgin soil.
6. XRD analysis clearly confirmed the improvements gained in the mechanical and engineering performances of the soil stabilised with TBCB. The formation of cementitious hydrates such as C-Al/K-S-H, Portlandite (CH) and ettringite, were evidenced using XRD and SEM techniques. The results obtained were found to be consistent with the developments achieved in the compressive strength of the soil after treatment with TBCB. The morphological components of the hydrated paste of TBCB were found comparable to that for the RF over similar curing times. Therefore, it can be concluded that the formation of cementitious products contributed to the development of the UCS of the soft soil stabilised with TBCB.

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