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Incorporation of a nanotechnology-based product in cementitious binders for sustainable mitigation of sulphate-induced heaving of stabilised soils

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ABSTRACT

Sulphate-induced heaving in soils is a common problem caused mostly by the use of calcium-based binders in the stabilisation of sulphate-bearing soils. Sulphate-induced heaving is attributed to precipitation and growth of ettringite minerals in a high alkaline environment. A sustainable means of reducing sulphate-induced heaving by the addition of an additive called "RoadCem" (RC) to soils stabilised by calcium-based cementitious products has not been studied. RoadCem (RC) is an additive that is manufactured based on nanotechnology and comprises synthetic zeolite, alkaline metals and some complex activators as some of its constituents. This research is therefore concerned with the performance of a sulphate-bearing soil stabilised by partially substituting cement (CEM I) with ground granulated blast furnace slag (GGBS) and incorporation of marginal quantities of RC. Laboratory studies including oedometer free swelling testing, unconfined compressive tests and microstructural analyses of the phases of hydration in the stabilised soils were carried out. Results indicated a reduction in heave by about 67% when 1% of RC was included in the cementitious mix with 50% of the CEM I replaced by a combination of GGBS and RC. The use of RC in the stabilised soil was even more promising than that in which only GGBS was utilised to replace half of the CEM I proportion in the stabilised soil with the result indicating almost a 30% difference in heave reduction. Results also indicated an increase in the unsoaked strength of stabilised soil with RC inclusion compared to the stabilised mix without RC. Scanning electron micrograph studies revealed almost a complete elimination of heave-causing ettringite minerals under a 28-day hydration phase of the stabilised soil when using RC.

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1. Introduction

Sulphates in soils are either inherently a part of the natural soil or are imparted by secondary sources. Hydrated calcium sulphate or gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$), sodium sulphate or Glauber's salt (Na_2SO_4), and magnesium sulphate or epsomite (MgSO_4) are some of the commonly documented sulphates that could occur primarily in the soil in its natural form [1]. On the other hand, groundwater transportation or contaminated water resulting from construction site activities, oxidation of pyrites (FeS_2), etc can all contribute to introduce sulphates into the soil and thus act as secondary sources [2,3].

Research has proven that the use of calcium-based stabilizers for the treatment of sulphate-rich expansive clays can result to unwanted heaving (otherwise known as sulphate-induced heaving) rather than reducing it [4,5]. As the name implies, this phenomenon of heaving is known to occur in the presence of sulphates in the natural expansive clay and is due to the utilization of stabilizing agent such as cement or lime in the soil improvement activity. Within the mechanism of soil-stabilizer reaction, a pH environment (between 11 and 13) does develops that leads to the formation of "ettringite" - an expansive mineral in the hydrated system. Ettringite is a hydrous calcium aluminosulphate mineral compound (typically needle-like crystals) that tends to precipitate in very high alkaline conditions in concrete and soil systems where high sulphate activities occur. The complex ettringite mineral compound ($\text{Ca}_6[\text{Al}(\text{OH})_6]_{20} \cdot 24\text{H}_2\text{O}$) (SO_4)₂ H₂O could contribute to the sulphate heaving distress in the soil-binder reaction system by hydration or by a progressive growth

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and development of itself [6]. Another type of mineral that could be found to exist in the presence of the expansive ettringite is “thaumasite”. The formation of thaumasite mineral $\{Ca_6 [Si(OH)_6]_2 (SO_4) (CO_3)_2 \cdot 24H_2O\}$ does occur due to low temperature (<15 °C) sulphate reaction and very intensive carbonation [7]. However, the degree of expansion resulting from ettringite is considered more than that of the thaumasite mineral. Moreover, the thaumasite is presumed to be made up of about 45% of the ettringite volume from which it is derived [8].

The sustainable reduction of heaving caused by sulphates has been studied even though some field case histories are somewhat rare in literature [9,10,19,20,11–18]. Wild et al. [10,11] suggested the partial substitution of lime by GGBS as a technique to suppress heaving by carrying out tests (unconfined compressive strength and swelling) to establish the right combination of the lime and GGBS required. The used GGBS contributed in the hydration process to reduce ettringite formation by providing more of the silicon and aluminium both of which would react with Ca^{2+} ions to produce complex cementing gels. It was also noticed that a very high replacement ratio of the lime by GGBS (about 83%) would have to be used to enable an effective protection against sulphate heaving. However, Celik and Nalbantoglu [12] pointed out that this manner of mitigating the attacks of sulphate must be adopted with caution in order not to risk a further provision of a conducive environment for ettringite formation resulting from extra amounts of oxides remaining in the hydration phases of the treated soil.

Based on the foregoing, a nanotechnology-based additive called “RoadCem (RC)” is proposed in this research to aid a reduction in sulphate-induced swelling in a stabilised soil system that is composed of CEM I and GGBS. RC is an industrially-developed additive blend composed mainly of synthetic zeolites and alkaline metals with complementary activators to enhance its unique qualities. It is important to note that the process of production or manufacture of RC is “based” on nanotechnology. Hence, the actual particle sizes of the product may be larger than most nanosized particles used in soil stabilisation as reported in recent researches [21–25]. RC has also been reported as being environmentally-friendly as well as possessing great economic benefits when used in construction [26,27]. Therefore, an inclusion of marginal quantities of RC to stabilised sulphate-rich soils could mean that the high substitution ratios of a calcium-based binder with GGBS (about 83%) as stated in previous studies can be further reduced. This study proposes to reduce the proportion of CEM I by 50% and hence the CEM I replacement ratio with GGBS by an addition of 1% of RC in the soil-binder mixtures. Hence, an investigation into the notable sustainability credentials of GGBS, used as a partial substitution for CEM I with a further incorporation of RC for the purpose of sulphate-induced expansion mitigation in a stabilised soil system is the main thrust for this original research.

2. Materials and methods

2.1. Soil

Industrially processed kaolinite was used as the soil material in this research. The powdered material was supplied by Mistral Industrial Chemicals located in Northern Ireland, United Kingdom (UK). Unlike the naturally occurring clay, the industrial processing of the kaolinite ensures that it is “pure” and of known composition because some organics, sulphates or sulphides and other trace compounds would have been skimmed off. This enables the micro-structural, physico-chemical features and behaviour of the stabilised clay to be ascribed to the true composition of the mixture and the testing conditions adopted. Moreover, according to Knopp and Moormann and Rollings et al. [5,28], kaolinite is chem-

ically very rich in alumina as compared to most expansive minerals because it can give up more alumina in high pH-controlled environmental conditions to participate in the formation of expansive ettringite minerals and thus becomes more susceptible to attacks by sulphates.

2.2. Sulphate soil

In order to simulate the presence of sulphates, the natural soil samples were doped with varying predetermined proportions (4%, 8% and 12% by dry mass of soil) of hydrated calcium sulphate or gypsum ($CaSO_4 \cdot 2H_2O$). The gypsum was sourced from APC pure located in Cheshire, United Kingdom. This brand of processed gypsum has a purity of 99.0%. The nomenclature “S” will be adopted subsequently to represent the sulphate-bearing soil.

2.3. Binders

The CEM I utilized was supplied by Hanson Heidelberg group in UK and was produced to comply with BS EN 197-1 (2011) requirements of Portland cement (CEM I) having a strength class of 52.5 N which assures rapid setting and hardening in cold weather works. The GGBS used in this study was manufactured and tested by Hanson Heidelberg cement group in compliance with the requirements of BS EN 196-2 (2013). The RoadCem additive used is based on synthetic zeolites (5–10% by mass), alkali earth metals (60–80% by mass) and complemented by some activators (5–10% by mass). This RC was supplied by PowerCem Technologies in Moerdijk, The Netherlands. The chemical compositions of the used soil and binder materials are provided in Table 1.

2.4. Sulphate soil-binder combination programme

The natural sulphate soil (soil-gypsum mix) was stabilised by 8% of the CEM I binder calculated by dry weight of the soil. The chosen quantity of CEM I is in agreement with some established procedures and standard recommendations for stabilisation of the type of natural soil used in this study [29–33]. 1–2% (by dry weight of CEM I) of RC is usually recommended industrially for combination with CEM I for the purpose of soil stabilisation [34,35]. Given that the aim of this research is to reduce CEM I by 50%, GGBS which is an environmentally-friendly cementitious by-product was utilized to partially replace the CEM I while preserving the recommended RC quantity of 1%. The effect of RC in the stabilised soil were investigated by comparing the soil-binder mixtures with and without the RC included. Table 2 shows the binders used and the mix design adopted while Table 3 presents further detail of the soil-binder design proportions including their nomenclature.

2.5. Material preparation and testing

2.5.1. Index property testing

The basic geotechnical properties of the kaolinite were determined based on the ASTM standard methods of testing as presented in Table 4. Laser diffraction technology (using the Malvern Mastersizer 2000) was carried out as outlined in Eyo et al. [36] to analyse the grain sizes of soil and binders in their dry states and given in Fig. 1. Kaolinite is slightly uniformly graded while the binders or additives as could be seen are poorly graded. Both CEM I and GGBS seem to cut across both the silty and clay particle sizes ranges (0.0001 – 0.1 mm). This also invariably means that 100 percent of both CEM I and GGBS are finer than the RC on a sieve of 0.425 mm size (or sieve No. 40). On the other hand, RC could be regarded as being somewhat gravelly sand considering the range of particle sizes it includes (i.e. approximately between 0.1 and

Table 1
Chemical composition of soil and binders.

Oxide	Kaolinite (%)	CEM I (%)	GGBS (%)	RC ¹ (%)
SiO ₂	49	20.7	34.1	21.2
Al ₂ O ₃	36	4.6	13	1.7
Fe ₂ O ₃	0.75	2.3	0.51	0.63
CaO	0.06	64	39	47.1
MgO	0.3	1.7	9.5	4
K ₂ O	1.85	0.4	0.5	7.46
TiO ₂	0.02	0.3	1.3	-
Na ₂ O	0.1	0.1	0.3	-
SO ₃	-	2.9	0.3	-
Mn ₂ O ₃	-	0.1	0.7	-
LOI	12	2.9	1.9	-

¹The oxide component not included in the table is H₂O which is 17.9 for RC.

Table 2
Binder mix design.

Binder	Designation	Replacement ratio (%)	Total binder % by dry wt. of soil
Cement	C	100	8
Cement: GGBS	C-GGBS	50:50	8
Cement: GGBS: RoadCem	C-GGBS-RC	50:49:01	8

Table 3
CEM I replacement mix proportion.

Mix designation	Gypsum (sulphate) content (by dry wt. of soil) (%)	Binders in the mix
S0	0	None
S4	4	
S8	8	
S12	12	
S0 - C	0	CEM I
S4 - C	4	
S8 - C	8	
S12 - C	12	
S0 - C - GGBS	0	CEM I + GGBS
S4 - C - GGBS	4	
S8 - C - GGBS	8	
S12 - C - GGBS	12	
S0 - C - GGBS - RC	0	CEM I + GGBS + RoadCem
S4 - C - GGBS - RC	4	
S8 - C - GGBS - RC	8	
S12 - C - GGBS - RC	12	

10 mm size ranges). The moisture contents of the samples used in the subsequent performance of the engineering tests were determined at optimum conditions in accordance to ASTM D1557-12e1 (2012).

Table 4
Properties of kaolinite.

Property	LL	PL	PI	SC (%)	CC	SG	MDD	OMC	USCS	Free swell
				(%)	(%)		(kN/m ³)	(%)		(%)
Value	58	30	28	74	26	2.6	15	17	CL	12.6
Standard	ASTM D4318-17 (2017)			ASTM D422-63 (2007)		ASTM D854-10 (2010)	ASTM D1557-12e1 (2012)			ASTM D4546-14e1 (2014)

LL = liquid limit; PL = Plastic limit; PI = Plasticity limit; SG = Specific gravity; CC = Clay content; SC = Silt content; MDD = Maximum dry density; OMC = Optimum moisture content; USCS = Unified soil classification system.

2.5.2. Soil-binder preparation

All the materials (soil and binders) used were present in powdered form hence, thorough manual mixing of the products for several minutes by hand and the use of laboratory spatula was necessary in order to ensure proper homogeneity. The amount of water added to soil-gypsum-binder system was determined based on the optimum moisture content (Table 4) of the natural soil derived from the standard proctor compaction test ASTM D1557-12e1 (2012) but with an additional 2% of water to accommodate for the binders used based on research and experience [11]. The compaction curve representing the relationship between optimum water content and maximum dry density for the soil is shown in Fig. 2. Further thorough mixing of the materials with water was carried out for about 10 min in order to prevent the development of 'hot spots' and non-uniformly migrated ions as well as improving the chances of uniform ettringite nucleation site distribution [37]. Extended mellowing was not carried out because the amount of sulphate used are estimated to be sufficient for the development of ettringite nucleation sites [37].

2.5.3. Solubility of gypsum

It has been established by research that the soluble sulphate threshold needed for sufficient ettringite formation and which may create a substantial risk of sulphate-induced damage is about 0.3% and above [18,38,39]. Gypsum has been known to have a solubility of 2.58 g per litre of water [8,40]. However, due to its relatively low solubility property, the amount of water that is required to dissolve gypsum becomes very crucial. One mole of gypsum is known to have a mass of about 172 g while containing approximately 96 g of SO₄. By using this analogy, if 100 g of dry soil contains about 0.3% (0.3 g) of gypsum or 0.167% sulphate and 22 g of water (which is the optimum moisture utilized for the mixtures in this research to accommodate the binders) then the amount of the water-soluble sulphates that can be solubilized is given by:

$$22g(H_2O) \times \frac{2.58g(gypsum)}{1000g(H_2O)} \times \frac{96g(SO_4)}{1mole(gypsum)} \times \frac{1mole(gypsum)}{172g(gypsum)}$$

$$= 0.0317g(SO_4) = 0.0317\%SO_4$$

It could then be seen that the optimum moisture used as derived from the proctor test only permits about 0.0317% of soluble sulphate which is well below the threshold of 0.3% for sulphate-induced disruption as suggested in literature [18,38,39]. Hence, in order to allow for an adequate sulphate dissolution, total soaking with water of the compacted samples will have to be ensured as would be described subsequently during the actual heaving test.

2.5.4. Sample preservation and curing

After extrusion of the samples from the compaction mould using the extractor jack, they were wrapped in a cling film and further sealed in zip-lock type of bags and preserved in the bucket to cure for a period of up to 28 days at a temperature of 20 ± 2 °C.

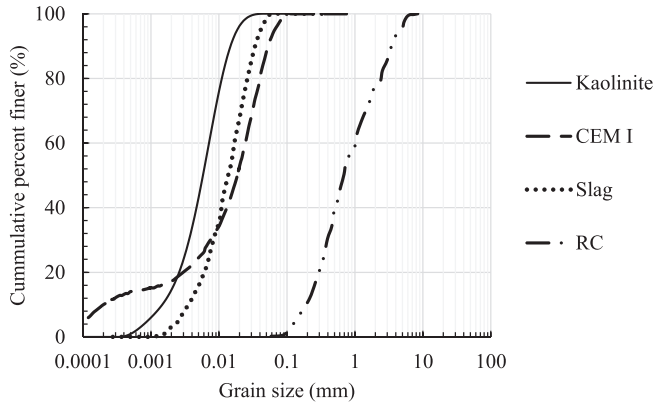


Fig. 1. Grain size of materials.

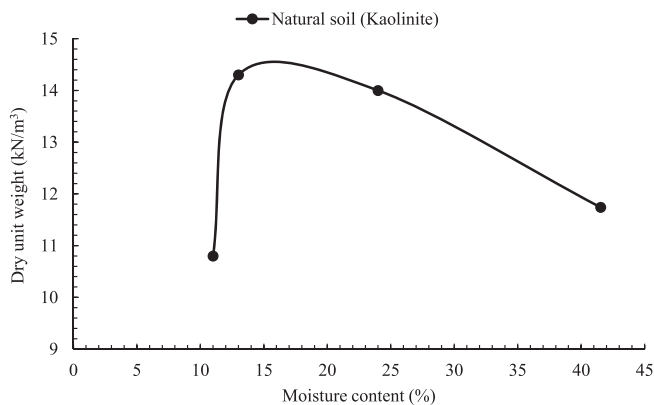


Fig. 2. Compaction curve of natural soil.

2.5.5. Oedometer swell test

The free swell oedometer testing procedure [ASTM D4546-14e1 (2014) Method A] which ensures that tested samples are completely soaked in water, was carried out on the cured samples. This method simulates field conditions where water replenishment from ground water flow and precipitation can result in constant dissolution of gypsum thus, providing a suitable environment for the formation and growth of ettringite [5,8,18]. Moreover, molar volume calculations have shown that water migration from an external source into a system of stabilised soil after thorough mixing and compaction, can be responsible for huge amounts of volumetric expansion in the layer of stabilised sulphate-bearing soils [37]. Before the free-swell testing, grease-coated standard oedometer rings measuring 70 mm in diameter and 20 mm in height were inserted into the soil and soil-binder materials in the standard proctor compaction mould and compaction carried in accordance to ASTM D1557-12e1 (2012) with the rings in the mould. This was done in order to eliminate the problem of breakage or cracking upon coring after compaction and extraction of the samples from the mould. The extracted material was carefully trimmed to remove the soil or soil-binder-laden oedometer ring. At the completion of the designated periods of curing, the ringed samples were transferred to the consolidation apparatus, inundated and allowed to swell under a seating pressure of 5 kPa.

2.5.6. Unconfined compression test (UCS)

This test was performed in accordance with ASTM D2166-00 (2000) on the compacted samples but with the samples unsoaked. Grease-coated standard cylindrical steel of dimensions 76 mm

height and 38 mm diameter were cored through the samples compacted using the standard proctor mould as described in ASTM D1557-12e1 (2012) and the cored samples extracted and preserved. The UCS test was conducted using an automated triaxial machine with the horizontal or lateral stress maintained at zero through the elimination of the cell pressure ($\sigma_3 = 0$) which is normally provided by water. According to the test standard, the rate of strain may not exceed 2.0%/min of the original height of the test sample. In adherence to this rule, a constant rate of axial displacement of 1.0 mm/min was followed in this study. In order to account for any possible swell on the compacted samples treated with sulphates (without soaking) given that the optimum moisture was used, the standard UCS test was conducted after 7 days of curing to ascertain the effect of sulphate on strength.

2.5.7. Sample size

The typical standard tests adopted (oedometer and unconfined compression testing) usually require a good number of samples to be prepared. In order to minimise errors due to the sample size used, for the unconfined compression test, three samples (at most) were used for each test and the average value derived for each data points. The oedometer test utilised two samples each. Hence, by also considering that 7- and 28-days curing had to be completed before the respective testing, a total of 98 compacted samples were prepared and tested.

2.5.8. Micro-structural observation

Image analysis using the scanning electron micrograph (SEM) was conducted in order to provide a description of the mechanism of change in the fabric of the samples of natural and stabilised soils. Small-sized chunks derived from selected originally compacted and cured samples were used to obtain the micrographs. The samples were initially left to air dry before the SEM measurement. Also, to ensure that the surfaces of the samples were sufficiently conductive electrically before SEM measurements, a Polaron SC7640 sputter coater was used to coat the samples with gold. The ZEISS EVO equipment was then utilised to collect the micrographs (SEM) of the cured, dry and completely vacuumed samples. A minimum working distance (WD) of 7.7 mm utilizing a minimum acceleration voltage (EHT) of 5.00 kV and different degrees of magnifications to achieve clear images were observed.

3. Analysis of test results

3.1. Swelling of stabilised kaolinite

As was observed in Table 4, the natural kaolinite (without the gypsum added, that is "S0") has a maximum equilibrium swell value of 12.6%. However, it is noticed from as could be observed in Fig. 3 that the linear swell at equilibrium for the stabilised kaolinite are below that of the natural kaolinite after 7- and 28-days. Over the curing duration of 7 days, the soil stabilised by CEM I-GGBS binder combinations tends to swell the most whereas there is only but a slight difference in the final swelling at equilibrium between the soil stabilised by using CEM I alone and that in which CEM I is replaced by the by-product with the incorporated RC. An extension of the period of curing to 28 days means that the CEM I-stabilised soil would have the largest swell but with the soil stabilised by RC inclusion possessing the least amount of linear swelling (at almost zero percent).

3.2. Swelling of stabilised sulphate soil

Swelling of the natural sulphate-rich clays having different proportions of gypsum cured for 7 and 28 days are shown plotted in

Fig. 4. The percentage of swell of soils enriched by the sulphates seem to be more than the pure natural soil under 7 days of curing. However, as the curing period increased to 28 days, the swelling did appear to reduce as compared to the pure natural soil. Generally, the variation in swelling seems to be marginal and does not correspond to the proportion of gypsum added.

Fig. 5 shows the sulphate soils stabilised by various proportions of the binder combinations. Generally, the stabilised sulphate-rich soils all seem to swell higher than their stabilised counterparts without the gypsum added. It could also be noticed that the increase in the amount of swelling does not appear to correspond with the increase in the quantity of gypsum added for 7 days curing duration. In fact, the reverse seems to be the case as the 7-day cured samples containing the lowest quantity of gypsum (4%) tend to swell more than the samples having supposedly higher quantities of the sulphates. However, for the 28-day cured samples (with the exception of natural samples, the rise in the level of sulphate is consistent with the amount of swell in the samples stabilised with the binders and their combinations.

Investigation of the effect of the type of binders used and the curing duration are further carried out based on Fig. 6 which indicates the maximum swell percent values at equilibrium. Stabilisation of the sulphate-enriched soils generally acted to reduce the percentage of swelling with the passage of time (due to pozzolanic reactions) as compared to the pure natural soil. As also observed by Abdi et al. [41] the extended curing of a treated or stabilised

sulphate-bearing soil even before inundation can reduce swelling compared to the natural (non-sulphate bearing) soil. However, the effect of sulphate heaving can be appreciated by an examination of the adopted durations of curing (7 & 28 days) of the stabilised soaked sulphate-bearing samples.

Firstly, the heaving of the unstabilised sulphate-rich soils are noticed to be reducing with the increase in curing from 7 to 28 days for all proportions (4, 8 & 12%) of the gypsum added as stated previously. Fig. 6a shows that for the stabilised soil containing 4% of gypsum, the percentage of swelling decreases with curing duration. At 7 days, the amount of swelling seems to be slightly lower for the CEM I stabilised sulphate-bearing soil mix as compared to those stabilised with 50% of CEM I replaced by either GGBS or GGBS and RC. But at 28 days curing, the effect of GGBS and RC tend to act to reduce the swelling further. On the other hand, with an increase in the quantity of gypsum to 8 and 12%, the heaving of CEM I stabilised sulphate-rich soil does seem to increase (Fig. 6b and c). Nevertheless, at 28 days of curing of the stabilised soaked sulphate-rich samples, heaving is seen to reduce with 50% of the CEM I replaced by the by-product additives but with the RC having the most influence as the stabilised soil containing it produces the lowest percentage of swelling.

3.3. Unsoaked strength of stabilised sulphate soil

As stated previously, strength measurements were carried out to account for any sort of expansion which may have affected the soil strength without soaking of the samples prepared at the optimum moisture value.

Fig. 7 shows peak values of unconfined compressive strength of the natural and stabilised soils having different proportions of gypsum. For the stabilised soil without the gypsum added, a progressive rise in the UCS is noticed but with the mix containing RC having the highest strength. Moreover, for the mixes with the natural soil enriched with 4% gypsum, there is an even greater percentage increase in the strength (approx. 140% for S4, 165% for S4 - C, 26% for S4 - C - GGBS and 80% for S4 - C - GGBS - RC) compared to the mixes without gypsum. Again, Fig. 7a indicates that the highest strength is obtained when RC additive is added to the mix. The same trend of the rise in the UCS with the sulphate-enriched soil are observed in Fig. 7b and c with 8% and 12% of gypsum in the soil respectively. It is however evident that an increase in the amount of sulphate in the soil does not correspond to sufficient strength increase. Although, the strength increase seems to be only slightly apparent in the sulphate-enriched system with the CEM I binder added as the gypsum levels increases to 8 and 12%. In general, it appears the presence of sulphate in the compacted (unsoaked) soil and soil-binder system does aid the enhancement of strength.

4. Discussion

In order to enable an assessment of the mechanism of changes that occur in both the compacted natural sulphate and stabilised soils, SEM images are provided for the hydration phases. Moreover, given that the proportion of gypsum used did not produce sufficient variation in the properties of the natural and stabilised soils, only the micrographs showing 8% of gypsum added are provided.

4.1. Swelling of stabilised kaolinite

As was observed in Table 4, the natural soil (S0) or kaolinite possessed the greatest potential to swell. The compacted kaolinite has a leaf-like arrangement as seen in the SEM image presented in Fig. 8a. This microstructure suggests interlinked pore structure

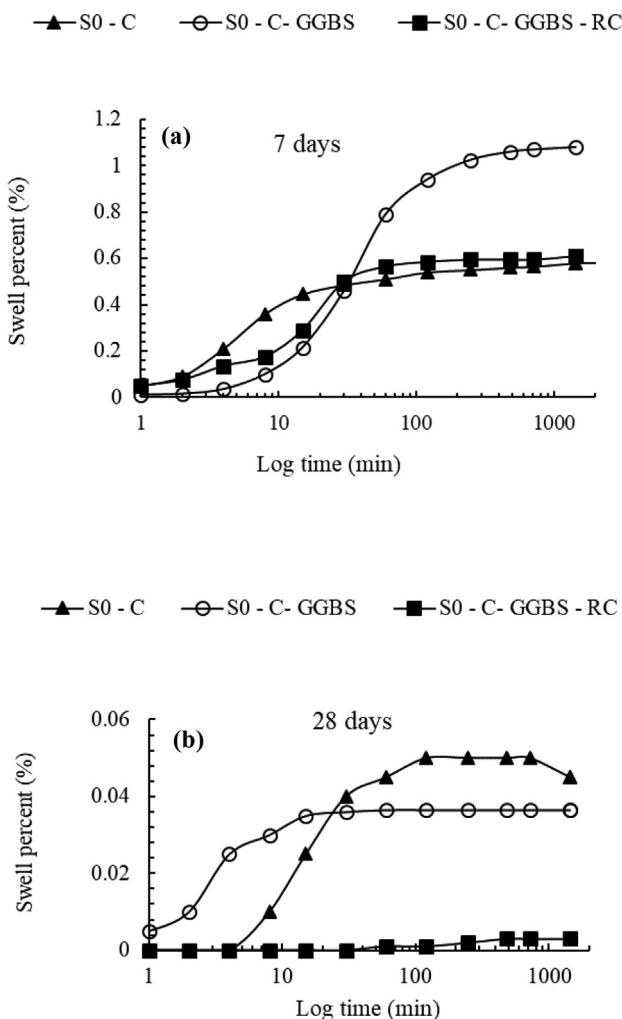


Fig. 3. Stabilised non-sulphate soil (a) 7-day cured (b) 28-day cured.

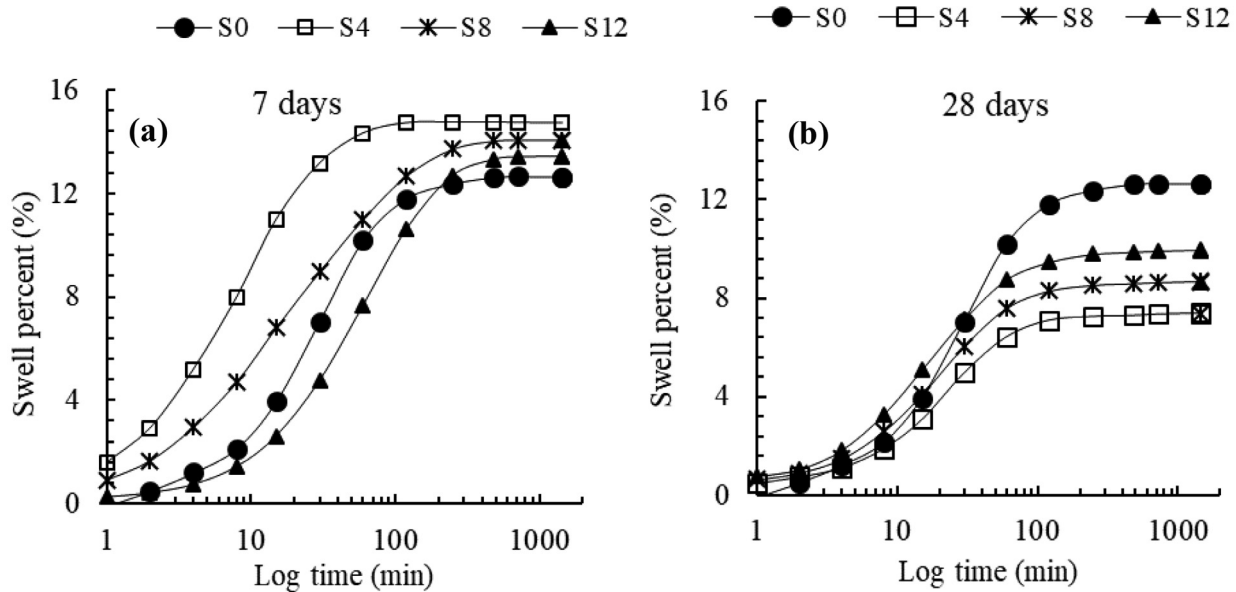


Fig. 4. Swell percent of natural sulphate soil with various gypsum content (a) 7 days curing (b) 28 days curing.

that can result in high rate of permeability during the initial and primary stages of swelling but reduced time of swelling when inundated with water. It is known that when soils rich in kaolinite are exposed to sufficient quantities of moisture, they tend to demonstrate an aspect of expansion known as inter-crystalline swelling [42,43]. The negative charges at the surface of the kaolinites would strongly attract water molecules (which has a relatively thin monolayer thickness) to themselves. This phenomenon then leads to the formation of an extensive adsorbed film as a result of the concentration gradient that exists between the electrical double-diffused layer (composed of molecules of water and some exchangeable cations) and the bulk solution. Stabilisation by the cement and slag binders used in this study can modify the created electrically charged double layer by reducing the existing adsorbed water layer thickness hence, leading to a reduction in the swelling potential of the soil. The mechanism of reaction that results from the use of a single calcium-based binder (such as CEM I or GGBS) in soil stabilisation is well-known [44,45]. However, the mechanism of reactions that ensues using both GGBS and CEM I to stabilise a soil has two fundamental phases namely, hydration of GGBS by hydrated lime from the CEM I and soil-hydrated lime reactions. Firstly, the hydration of GGBS proceeds with the consumption of very little amounts of lime and commences soon after water is introduced and used to mix the soil- binder materials. This reaction tends to lead to the production of calcium aluminosilicate hydrates (CASH) having low calcium to silicon ion ratio, aluminium to silicon ion ratio and calcium to aluminosilicate ions ratio. The second phase involves the soil-CEM I (or hydrated lime from CEM I) reaction and leads to the production of colloidal CASH. These resulting crystalline products of hydration tend to proceed in a much slower rate than CEM I hydration and thus possesses some 'pore-blocking' effects and reduced double layer leading further to the increase in long-term hardening of CEM I paste and by extension an enhancement of the stabilised soil's engineering properties such as strength and swelling.

In the shorter term though (7 days of curing), the reduction in swell is mainly attributed to the decreased affinity of the soil particles to the absorb water however, as time progresses (say at 28 days of curing), the formation of crystalline pozzolanic reaction products (i.e. CAH, CASH or CSH) occurs which aids the further reduction in swelling (Fig. 8b & c) [41,46]. However, it has been

reported that the mechanism of cementitious binders (especially cement) reaction which produces complex hydrates having complete spherical barrier, would prevent further penetration and reaction of the binders [47]. But, RC addition to cementitious binders allows for deeper penetration of both it and the water of hydration through the breakage of the barrier formed by pozzolanic reaction products and a further increase in pH. As a result, a much larger amount of the water molecules is converted into crystalline water with more of the crystals occupying any spaces left in the process of hydration. It is this extended crystallization which is also accompanied by a sharp reduction in the heat produced in the hydration that ultimately enhances swell reduction and possibly strength increase in the soil-binder mix. Fig. 8d shows the encapsulation or 'wrapping' (interlocking matrix) structure formed from the crystalline products in the soil-binder hydration mechanism which is as a result of the addition of RC to the cementitious materials. Similar observations were made by Ventura and Koloane and Eyo et al. [48,49] who investigated the addition of 1% RC to a soil-cement-fly ash system of mixes and a soil-cement-GGBS system respectively.

4.2. Sulphate soil swelling

Upon soaking of the compacted samples in water immediately after 7 days curing, Fig. 6 indicated some slight increase in swell of the non-stabilised sulphate-rich soil for all the amount of gypsum considered compared to the pure natural soil. As noticed previously in Fig. 7, the relatively stronger bonds formed due to the exchange occurring in the hydration process between the calcium ions (from the gypsum) and the weaker cations (from the soil), led to strength increase (of the compacted samples without inundation) of the sulphate rich soil. However, the same forces could not withstand the constant penetration of external water upon soaking within 7 days of curing. A similar expansion effect of the presence of sulphate in natural kaolinite was observed by Abdi et al. [41] who rather attributed the outcome to a slight decrease in the pH level (rise in acidity) of the soil-gypsum pore solution which engenders more adsorption of the water hence, resulting in increased swell potential.

Nevertheless, as the curing duration increased to 28 days, swelling of the sulphate-rich soil did appear to reduce compared to the

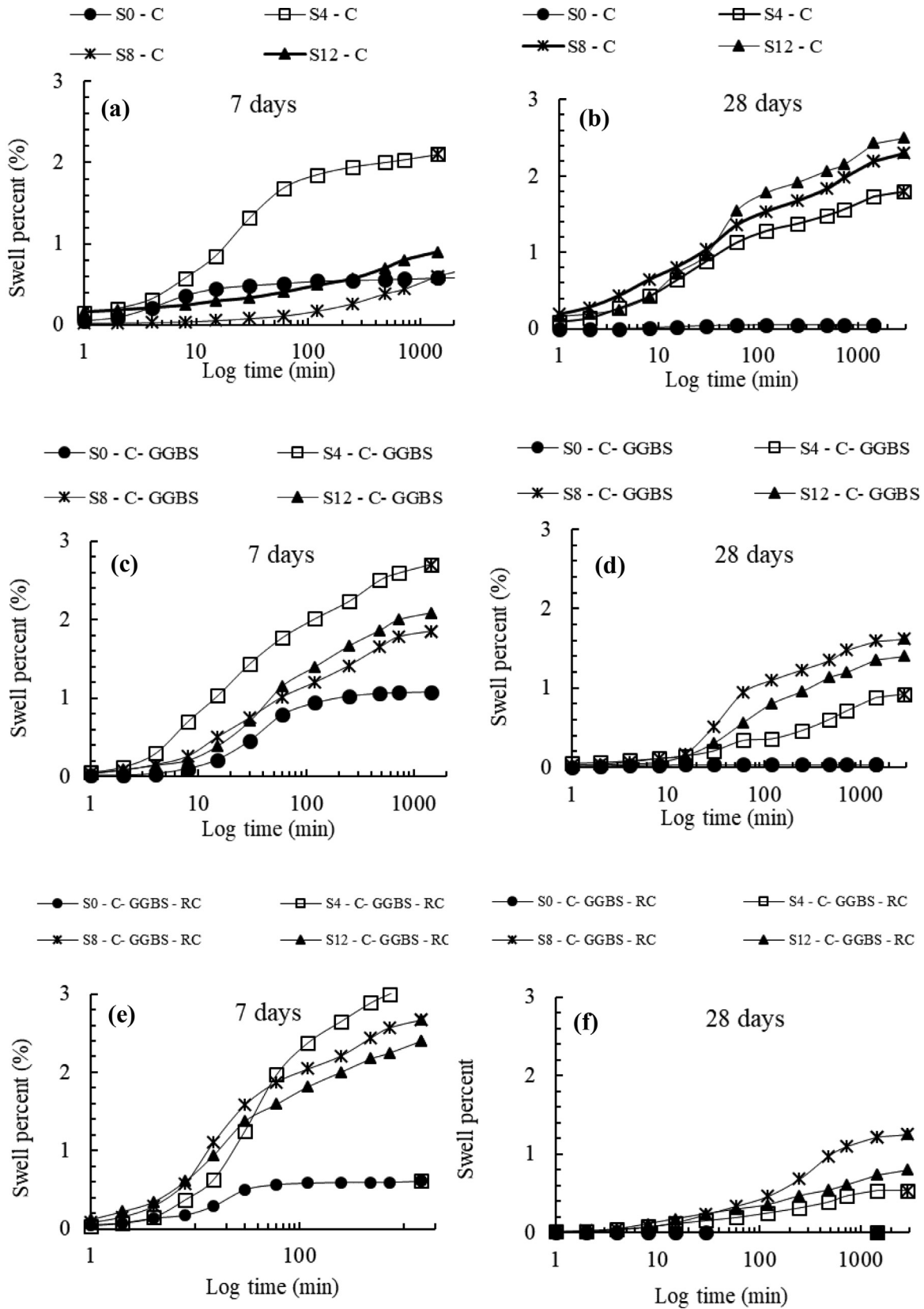


Fig. 5. Swell percent of stabilised sulphate soil with various gypsum content (a) C-stabilised soil at 7 days curing (b) C-stabilised soil at 28 days curing (c) C-GGBS-stabilised soil at 7 days curing (d) C-GGBS-stabilised soil at 28 days curing (e) C-GGBS-stabilised soil at 7 days curing (f) C-GGBS-stabilised soil at 28 days curing.

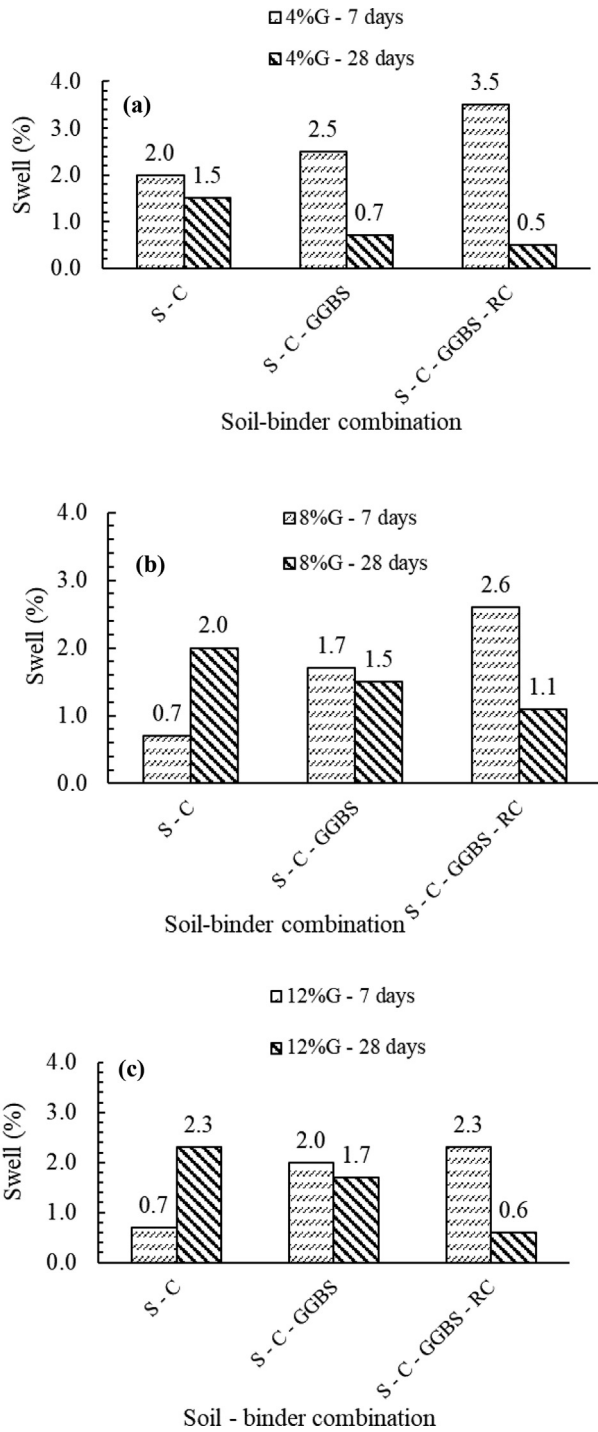


Fig. 6. Maximum swell percent of stabilised sulphate soil with various gypsum content (a) 4% gypsum (b) 8% gypsum (c) 12% gypsum.

pure natural soil (Fig. 4b) indicating that an extended period of time was indeed required for more hydration and formation of stronger bonds which acted to reduce the swelling.

4.3. Stabilised-sulphate soil swelling

The sulphate-rich soils stabilised by the binders and their various combinations all seemed to swell higher than the stabilised soils in the absence of sulphates at both 7 and 28 days of curing (Fig. 5). Prior to soaking, the sulphate ions and alumina existing

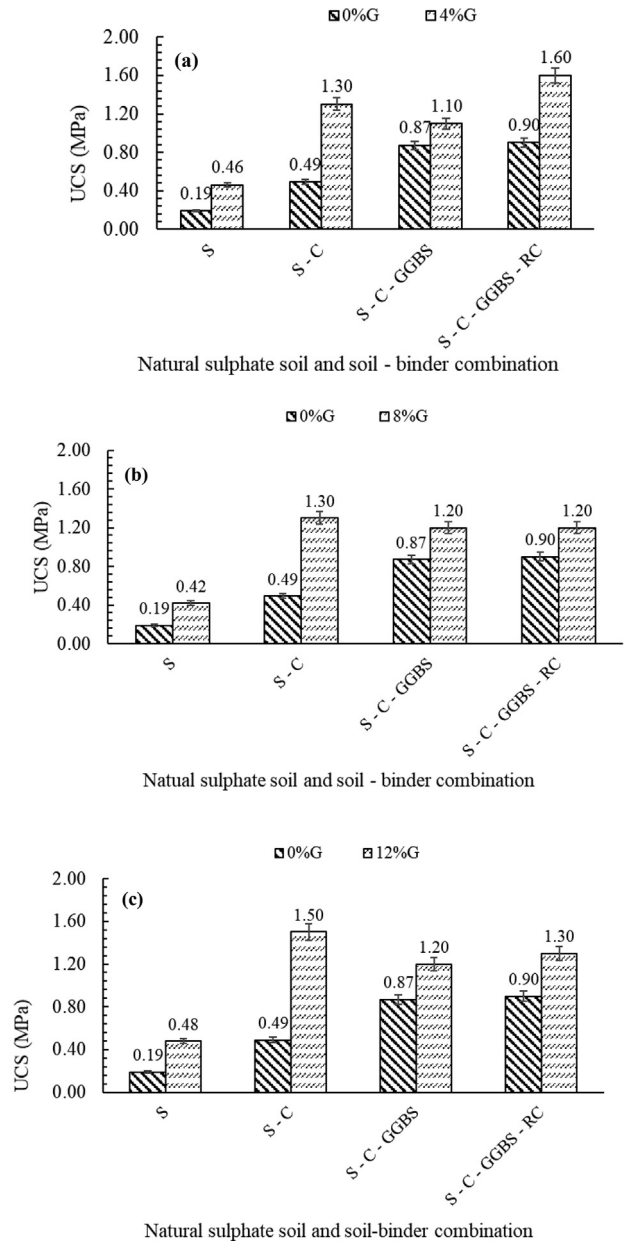


Fig. 7. Strength of stabilised soil (a) with 4% gypsum and without gypsum (b) with 8% gypsum and without gypsum (c) with 12% gypsum and without gypsum.

only in limited amounts in the stabilisation reactions would bring about the precipitation of ettringites which within this time only serve as nucleation sites for potential future growth of the crystals when both the sulphates and alumina become readily available [37]. Upon soaking, the movement of water into the stabilised sulphate will tend to solubilise the unreacted cementitious binders as well as acting as an avenue for more ionic migration hence, becoming a continuous source of supply of reagents at the nucleation sites [4,40]. At 7 days, the amount of swelling seemed to be the lowest for the CEM I stabilised samples (Fig. 6). Within this relatively short period, the initial hydration resulting in cationic exchanges and flocculation/agglomeration reactions may have been experienced more by the CEM I-stabilised sulphate soils as compared to the sulphate soil stabilised by the by-products regardless of the amount of gypsum used. However, with the curing extended to 28 days, the effect of the by-product additives (GGBS and RoadCem) tended to act to reduce the swelling more than

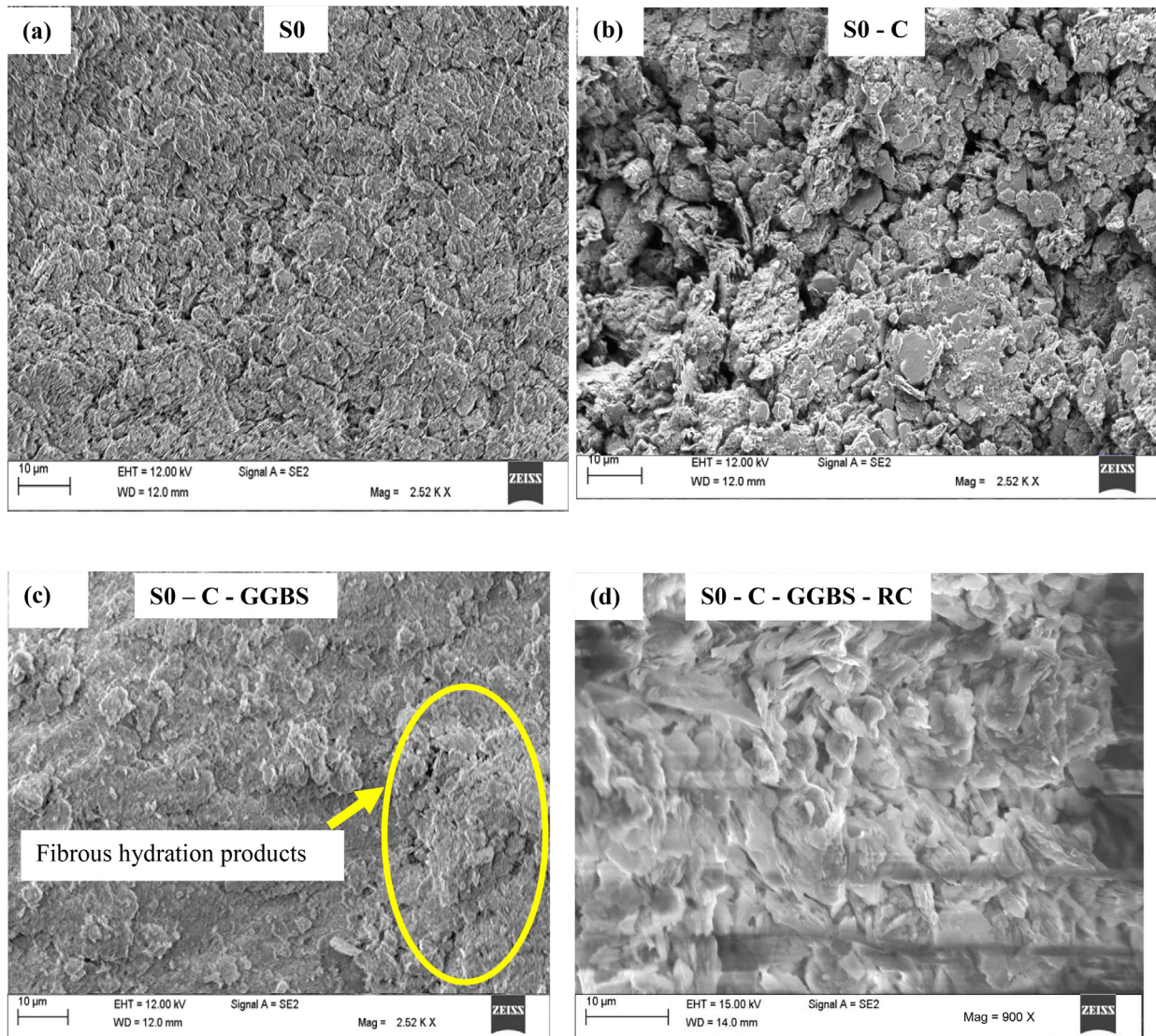


Fig. 8. Natural and stabilised non-sulphate soils (a) Natural soil (S0 without sulphate) (b) C-stabilised non-sulphate soil (c) C-GGBS- stabilised non-sulphate soil (d) C-GGBS-RC- stabilised non-sulphate soil.

the CEM I used alone. Fig. 9a shows the formed ettringites as a result of stabilisation of the sulphate soils by CEM I at 28 days curing upon soaking with water. As stated previously, it is known that for the sulphate soil-C-GGBS system, sets of reactions occurring either simultaneously or in lieu of the other will ensue [43]. The hydration of GGBS which is activated by CEM I in the presence of the sulphate results in the formation of C-S-H and ettringite products, soil-CEM I reaction in the presence of sulphates resulting in C-S-A-S-H and ettringite products as well as development of competing nucleation sites on the soil surfaces and the GGBS surfaces. For the partial replacement of CEM I by the GGBS alone without the RC added, it has been suggested that the GGBS tends to contribute more in the production of cementitious gels needed in hydration reaction mechanism to further reduce or in some instances stop the growth of ettringites as seen in Fig. 9b over 28 days curing upon water soaking. Moreover, the protective influence against the attack of sulphates can become very much intense when higher amounts (up to 80%) of the GGBS are used to replace CEM I [10,19]. However, as seen previously in Fig. 6, only 1% of the RC used in the stabilisation process would serve to mitigate swell further than the GGBS at 50% of the CEM I replaced. It is believed that RC addition

acts as a catalyst to further promote the above-mentioned hydration reactions involving CEM I and GGBS which does ultimately result to the prevention of swelling. The presence of more external water to the sample enables the formation of more crystalline pozzolanic products with the RC included thus eliminating any possibility of ettringite formation as observed in Fig. 9c for the stabilised soil having 8% of the gypsum at 28 days curing upon soaking in water.

4.4. Unsoaked strength

The presence of sulphates seemed to have caused an increase in UCS for all the percentages of gypsum added to the natural soil (Fig. 7). A similar trend was observed by Jha and Sivapullaiah and Yilmaz and Civelekoglu [50,52]. Given that gypsum contains much of the mineral calcium hydroxide, the process of ionic exchanges between the gypsum (Ca^{2+}) and the soil ions (aluminium and silicon) and the formation of hydration products [such as calcium silicate hydrate (CSH) or calcium aluminosilicate hydrate (CASH)] should result to a rise in strength. Yilmaz and Civelekoglu [52] indicated that increasing the amount of gypsum

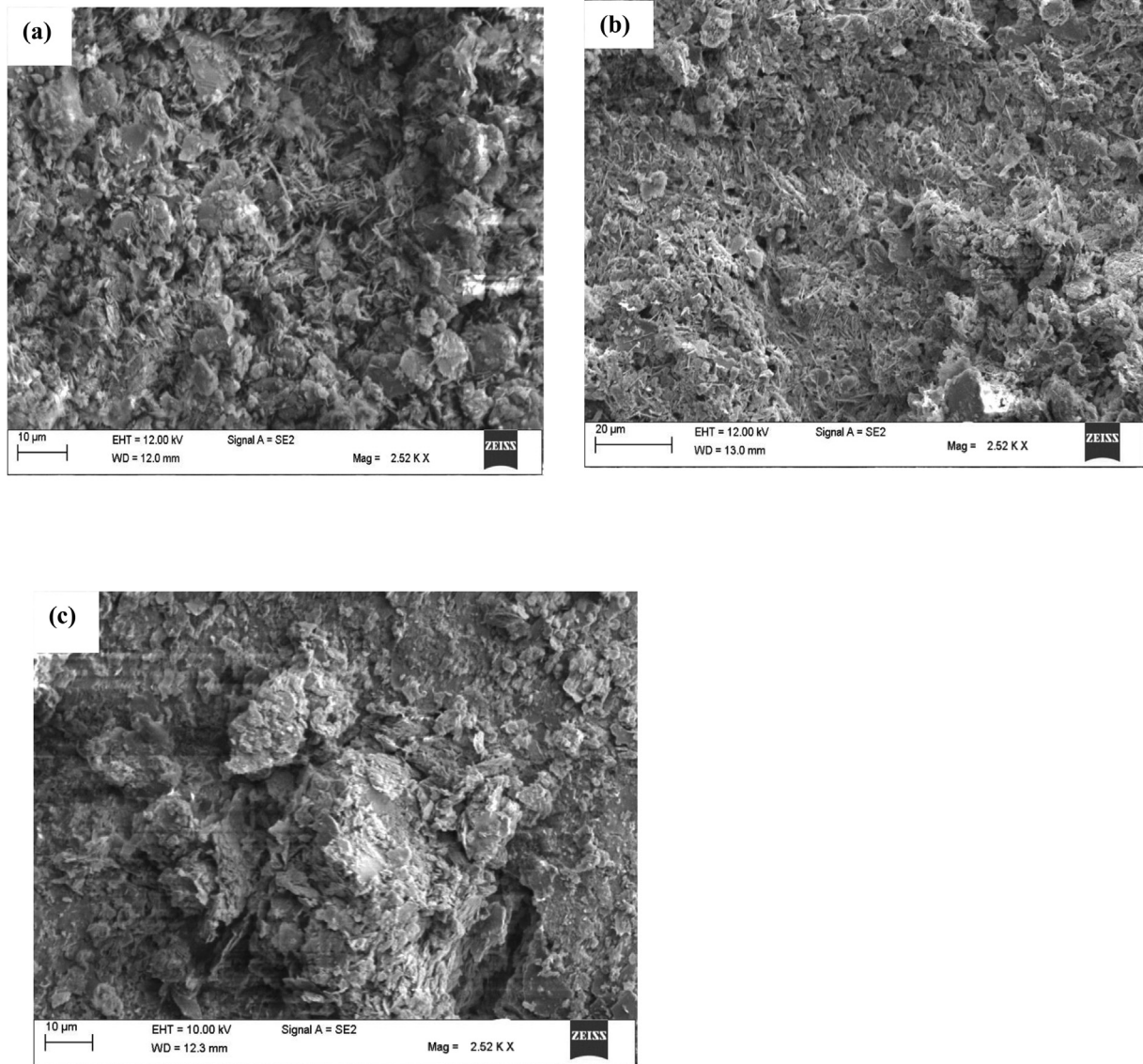


Fig. 9. Stabilised sulphate soil with the binder combinations (a) C-stabilised sulphate soil (b) C-GGBS-stabilised sulphate soil (c) C-GGBS-RC - stabilised sulphate soil.

in the natural soil beyond 5% may not result in significant increase in UCS. Hence, the increase in the quantity of gypsum from 4% to 12% as is in the case of this research did not result to much strength gain (Fig. 7). On the other hand, an even greater increase in the UCS of the sulphate-enriched soils were noticed when stabilised with the binders. It should be noted here that the compacted samples used for the UCS test were not soaked in water prior to the test. Except for the soil enriched by 4% of the gypsum, the stabilised sulphate soil consisting of CEM I used alone seems to have the highest strength. The additional supply of calcium ions to CEM I can result in strength increase for a short curing duration [51]. In this case, gypsum seems to act as a catalyst that accelerates the initial hydration rates of the stabilised soil leading to the formation of sufficient amount of the compounds of cementation (CSH and CASH). Moreover, the sulphate ion that is contained in the gypsum reacts with the alumina and calcium ions present in the soil and cement respectively to form the complex calcium silicate aluminate hydrate compound or the mineral called “ettringite” including the cementitious compounds of hydration (CSH and CASH) (Fig. 10b) [11,43,53]. The formation of ettringites aids the reinforcement and interlocking of the particles in the soil while the

produced cementitious compounds leads to the formation of a well bound cemented and compacted matrix that all contribute to increased strength [6,11,51]. In the case of the stabilised sulphate-C-GGBS-RC system, a combination of the multiple but sometimes competing mechanisms of hydration resulting from the sulphate soil-C-GGBS system with RC inclusion as mentioned previously, would lead to the production of more crystalline compounds of hydration through further and deeper penetration of the RC and water molecules of hydration to increase strength (Fig. 10d).

4.5. Effect of gypsum content

An increase in the amount of swelling did not appear to correspond to the increase in the quantity of gypsum added (Figs. 5 and 6). There could have been some limiting factors that did prevent the soils possessing higher quantities of sulphates or gypsum (8 and 12% of gypsum) from swelling linearly more than the soils with 4% of gypsum applied. As stated above, the mechanisms of ettringite formation (precipitated nucleation sites and subsequent crystal growth) tend to proceed very quickly the extent of which

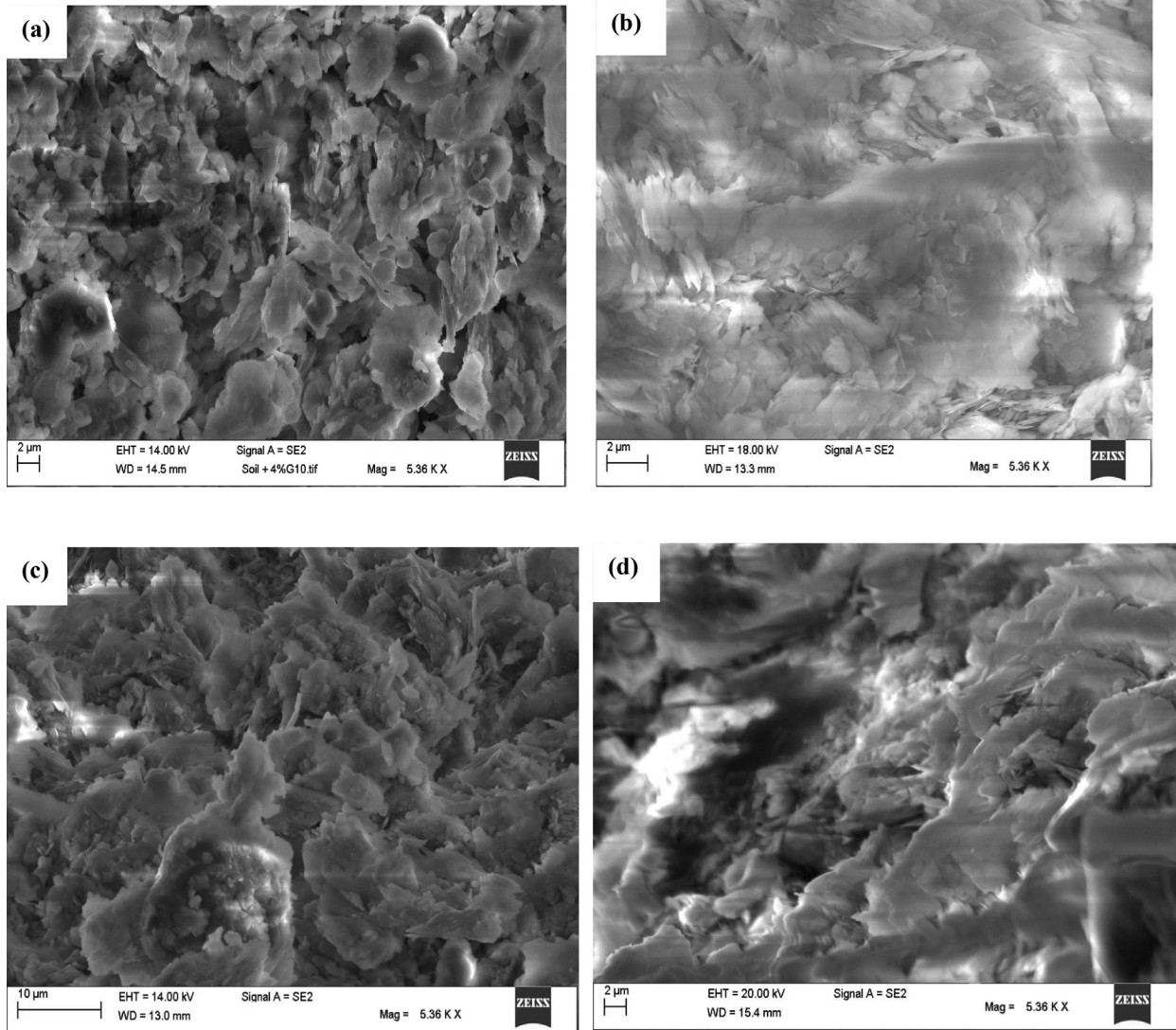


Fig. 10. SEM of natural soil and soil stabilised by binder combinations (a) natural sulphate soil (b) sulphate soil stabilised by C (c) sulphate soil stabilised by C-GGBS combination (d) sulphate soil stabilised by C-GGBS-RC binder combinations.

depends on the available limiting reagent present in solution [37]. Given that chemical reactions could only be judged as being as fast as their rate limiting steps (or their slowest steps), this limiting factor does manifests either at the interface of the solid-solution through a topochemical mechanism or at the through-solution (or hydration) mechanism depending on ettringites' formation mode [54,55]. The saturation level of the gypsum may have reached its limit at 4% (or slightly above that) or that the curing duration was relatively short, to allow for the completion of the process that would bring about an increased quantity of soluble sulphates. James [56] did suggest that the nature of the dissolved alumina and hydroxide ions may have also been the cause. With respect to the curing duration, it has been suggested that the process of ettringite formation that begins with the development of nucleation sites and subsequent growth of the crystal is time dependent [8]. As was noticed in Fig. 6b & c at 28 days, the rise in the level of gypsum (or sulphate) added seemed to be slightly consistent with the amount of swell expected in the samples stabilised with the binders and their combinations. The higher gypsum content in the soil in this case provides a continuous source of the sulphates for the formation of greater amount of ettringite. But again, this could be also dependent on other factors such as

the soil structure, as well as those stated above regarding the formation mechanisms.

5. Conclusion

The effect of RC on soil exposed to sulphate attack was investigated in this study. Overall, it can be concluded that the use of RC additive can be potentially effective for the mitigation of heave in sulphate soils stabilised by calcium-based binders. Even though further studies can be carried out by simulating the influence of various other environmental conditions, the results obtained in this research suggest a possible use of RC in cementitious binders for soil stabilisation. Some of the findings are as follows:

1. Swelling reduced significantly with curing time for the natural (non-sulphate-bearing) soil stabilised by an inclusion of RC to the cementitious mixture containing CEM I and GGBS. The effect of RC was clearly noticeable with the swelling reduced to approximately 0% after 28 days of curing under inundation.
2. The percentage of swelling of sulphate-bearing soil was slightly more (about 10% on average) than that of the natural (non-sulphate-bearing) soil at 7 days of curing. However, with the

curing period increased to 28 days, swelling did appear to reduce (approximately 30% on average) compared to the natural (non-sulphate-bearing) soil. Generally, the variation in swelling seems to be marginal and does not correspond to the amount or proportion of sulphates.

3. The stabilised sulphate-rich soils generally expanded more than the stabilised non-sulphate soils. In this instance, the rise in the level of sulphate was seen to be consistent with the amount of swelling in the samples stabilised with the binders and their combinations.
4. A reduction of about 53% in soil swelling was observed when GGBS was partly replaced by CEM I used to stabilise the sulphate-bearing soil compared to CEM I used alone. The GGBS contributed more in the production of the cementitious gels needed in hydration reaction mechanism to further reduce ettringite formation as compared to CEM I used alone in the soil stabilisation.
5. Inclusion of RC to the cementitious binders (including CEM I and GGBS) in the stabilised soil served to reduce swelling even further by approximately 29% compared to both CEM I and GGBS used together in the stabilised the sulphate-rich soil. More so, addition of RC to the cementitious materials reduced swelling by 67% compared to CEM I used alone to stabilise the sulphate-bearing soil. The presence of RC enabled the formation of more crystalline pozzolanic products thus eliminating any possibility of ettringite as observed from the SEM measurements.
6. Gypsum addition also resulted in an increase in the unsoaked strength of natural and stabilised soils.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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