



Article

# Strength and Swell Performance of High-Sulphate Kaolinite Clay Soil

Blessing Adeleke \*, John Kinuthia  and Jonathan Oti 

Faculty of Computing, School of Engineering, Engineering and Science, University of South Wales, Pontypridd CF37 1DL, UK; john.kinuthia@southwales.ac.uk (J.K.); Jonathan.oti@southwales.ac.uk (J.O.)

\* Correspondence: blessing.adeleke@southwales.ac.uk; Tel.: +44-1443-48-2333

Received: 17 November 2020; Accepted: 3 December 2020; Published: 5 December 2020



**Abstract:** Expansion of soils has been found to produce significant negative economic and environmental impact on various civil engineering infrastructure. This impact is more deleterious in soils containing sulphates, when treated with calcium-based stabilizers such as Lime and/or Portland cement (PC). The reported study investigated the strength and swell characteristics of Kaolinite clay artificially induced with high levels of Gypsum (sulphate) contents after stabilization with CEM I (PC), which is a calcium-based stabilizer. An optimum stabilizer content/Gypsum dosage, aimed at investigating the maximum magnitude of expansion possible using high levels of 10, 15 and 20% Gypsum contents (4.7, 7 and 9.3 wt.% sulphate) stabilized with calcium-based content of 7, 8, 9 and 10 wt.%. This was expected to provide further understanding on the mechanisms behind high sulphate-bearing clay soils, and the impact of sulphate and calcium content on strength and swell characteristics. The research outcomes showed that the introduction of sulphate to a Kaolinite clay soil reduces the compressive strength of the stabilised product by a factor range of 6–47% at 28 days curing age, while the swell behaviour is mainly dependent on both the sulphate content and curing age. Furthermore, the observed result suggests an 8 wt.% binder content to produce maximum magnitude of expansion (swell) with a high Gypsum content of 10% by weight. This finding is of economic importance, as it is expected to serve as a benchmark for further research on the stabilized clay systems, at high sulphate levels using sustainable binder materials.

**Keywords:** sulphate bearing soil; soil stabilization; Kaolinite clay; optimum sulphate content; mechanical strength; swell behaviour; linear expansion

## 1. Introduction

Swelling in soils is a three-dimensional process, which occurs when the fine particles of a soil material undergo a volumetric increase in size due to the absorption of water from its surrounding. The swelling is as a result of the incessant changes or fluctuation in moisture content caused by unstable seasonal weather conditions and flooding [1,2]. This volumetric increase in size is of key importance to the civil engineering industry based on the generation of swelling and large magnitudes of swelling pressure, which leads to the destruction and additional refurbishment cost to structures (building foundations, rail tracks, highway pavements, airports runways, tunnels, pipes, bridges, seaports etc) constructed in and on the soil [1,3].

Stabilization of soils has been found to be economically and technically effective in reducing swellings in expansive soils by chemically altering the properties of the soil, which improves on geotechnical and engineering properties of the stabilized/treated soil using Portland cement and Lime (Calcium based stabilizers) as activators [4–8]. Seco et al. [9]; Cheng and Heidari [10] and Schanz et al. [11] all attributed this swelling tendency to the mineralogical composition/physiochemical properties of the soil, type of clay with respect to Base Exchange Capacity (or cation exchange capacity), quantity of

clay, charge of exchangeable cations in the interlayer space, soil moisture content, plasticity and dry density and the type of material used in case of soil stabilization. A number of studies investigated and reported the reduction of this swelling tendency through the application of calcium-based materials (Lime and PC) for stabilization purposes. The application of calcium-based materials was also found to improve their mechanical performance, with the use and establishment of typical stabilizer dosage values (3–8 wt.%) for expansive soils [4,12–14].

However, many studies have also highlighted that sulphate-bearing soils are prone to strength loss, stability and durability risks due to the generation of expansive reactions, when treated or stabilized with calcium-based stabilizers [12,15–19]. These detrimental effects have been evidenced in natural sulphate-bearing soils and artificially dosed sulphate clay systems (Gypsum–Kaolinite clay). The increased expansion was believed to be partly caused by the formation of a highly expansive crystalline, and hydrated mineral from the hydration reaction of calcium (obtained from PC or Lime), alumina, silica, sulphate in the presence of water known as ettringite [ $\text{Ca}_6\text{Al}_2(\text{SO}_4)_3(\text{OH})_{12}\cdot 26\text{H}_2\text{O}$ ]. It is described as needle-like, with a large surface area and unsatisfied negative charge [20–22]. Research on the use of calcium-based material to stabilize sulphate bearing soils is insufficient. It was suggested that clay soils containing a mass fraction of at least 1% and 10% of sulphates produced significant swellings after treatment with calcium-based stabilizer, but with no emphasis on an optimum sulphate content [19,23,24]. Furthermore, Snedker and Temporal [25] identified the disastrous effects of lime stabilization during the construction of the M40 motorway near Banbury, U.K. This resulted in the formation of 60% heave of the stabilized capping layer, which contained 0.4 wt.% sulphates. Parker [26] also reported a 25% expansion of the carriageways on the 7.5 km A10 Wadesmill bypass U.K. Recently, a simulated sulphate-bearing clay systems was investigated by Diaz Caselles et al. [19] using 1 wt.% sulphate content to produce a 7% expansion when treated with lime. Li et al. [27] also developed an artificial sulphate soil system using Kaolinite clay, which was dosed with 2% sulphate content and stabilized with 10 wt.% of PC to produce a 6.1% expansion. Nevertheless, a full understanding of the mechanism behind the swelling tendencies, establishment of an optimum stabilizer content/sulphate dosage to produce optimum magnitudes of expansion at high sulphate levels has not been fully addressed.

Therefore, this paper presents the outcomes of a research study on the performance in strength and swell for non-sulphate bearing Kaolinite clay and an artificial sulphate soil system—Kaolinite clay dosed with high levels of Gypsum content (0, 10, 15 and 20 wt.%)—and stabilized with a calcium-based stabilizer (CEM I) within the range of 7–10 wt.%. This addressed the impact of sulphate and calcium content on strength and swell properties, provided additional information on the mechanisms behind CEM I–Kaolinite clay and CEM I–Kaolinite–Gypsum clay systems at high Gypsum dosages, and established an optimal sulphate content for an artificial sulphate soil system. This is expected to serve as a benchmark for further research on the stabilization of simulated sulphate-bearing Kaolinite clay systems at high sulphate levels using various sustainable binder materials.

## 2. Methodology

### 2.1. Materials

The materials used for this investigation were Portland cement (PC) (CEM 1-42.5N), Gypsum (GP), Kaolinite clay (K) and de-ionized water. PC was manufactured according to BS EN 197-1 [28] and supplied by Lafarge Cement UK [29]. The Gypsum used throughout this study was a white crystalline sulphate mineral that was manufactured by precipitation from aqueous solution, and supplied by Fisher Scientific in the form of calcium sulphate dihydrate ( $\text{CaSO}_4\cdot 2\text{H}_2\text{O}$ ). Kaolinite Clay (K) was a semi-processed industrial clay, processed and supplied by Imerys Minerals Ltd. UK, formerly known as English China Clay (ECC) International Ltd. UK. The main advantages of using semi-processed industrial Kaolinite clay include availability, consistency and homogeneity, and the relative ease of describing the reaction mechanisms before venturing into the more complex unprocessed clay soils. This enables the establishment of a more general, unbiased explanation of its performance and provides

a platform for the subsequent variations in other factors (e.g., moisture content and blending). Table 1 shows the chemical compositions for Kaolinite clay, CEM I and GP. The chemical compositions were obtained using a portable benchtop TXRF X-ray Fluorescence spectrometer, which is comprised of an air-cooled low power X-ray metal-ceramic tube with a molybdenum target. It runs at a max power of 50 W with a liquid nitrogen-free Silicon Drift Detector (SSD) [30,31].

**Table 1.** Chemical composition of Kaolinite clay, PC (CEM I) and Gypsum (GP).

Oxides	Composition (wt.%)		
	Kaolinite Clay	CEM I	GP
CaO	<0.01	61.49	32.81
SiO <sub>2</sub>	47.32	18.84	0.10
Al <sub>2</sub> O <sub>3</sub>	35.96	4.77	0.05
MgO	0.21	3.54	0.08
Fe <sub>2</sub> O <sub>3</sub>	0.69	2.87	0.03
Mn <sub>2</sub> O <sub>3</sub>	0.02	0.05	0.01
SO <sub>3</sub>	<0.01	3.12	47.31
TiO <sub>2</sub>	0.02	0.26	<0.01
K <sub>2</sub> O	1.80	0.57	<0.01
Na <sub>2</sub> O	0.07	0.02	<0.02
P <sub>2</sub> O <sub>5</sub>	0.12	0.10	0.03
V <sub>2</sub> O <sub>5</sub>	<0.01	0.06	<0.01
BaO	0.07	0.05	0.02
L.O.I.	13.10	4.30	19.90
Physical Properties			
Colour	White	Grey	White
Specific gravity	2.16	3.16	2.32

## 2.2. Mix Design, Test Sample Preparation and Testing

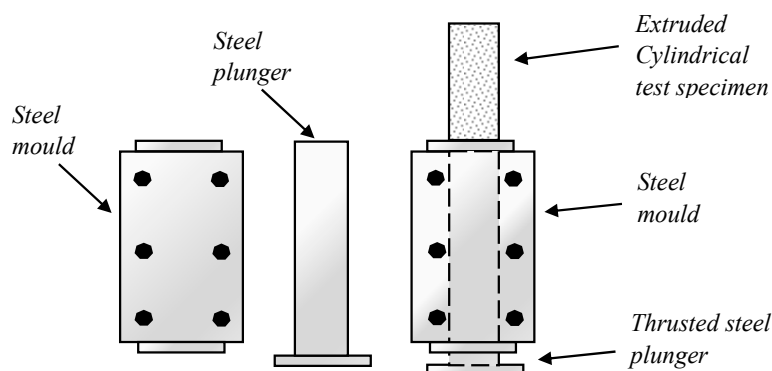
The mix design (see Table 2) was developed to produce cylinder specimens using varying compositions of CEM I, calcium-based stabilizer, with Gypsum dosed into the Kaolinite clay. This was achieved by utilizing various mix ratios of CEM I (7, 8, 9 and 10 wt.%) at 0, 10, 15 and 20% Gypsum content within the dry mass of Kaolinite clay. A total sulphate content (SO<sub>3</sub>) of 1.73 wt.% is equivalent to 3.72 wt.% by weight of Gypsum [20]. This indicates that the equivalent sulphate (SO<sub>3</sub>) content levels used in this study are 0, 4.7, 7 and 9.3 wt.%, respectively. The preparation of the cylindrical laboratory specimens was carried out for all the designed mix compositions in Table 2, and thus ensuring optimal compaction conditions with minor variations from the stabilized Kaolinite clay. These variations were kept under check, by ensuring that the experimental procedures were the same for all the cylinder specimens produced from the different mix compositions in each stabilizer dosage and Gypsum content group. Proctor Compaction tests were initially conducted in accordance with BS 1377-4 [32] to establish the Maximum Dry Density (MDD) and Optimum Moisture Content (OMC) values of the target material (Kaolinite clay) for sample preparation purposes.

The MDD and OMC values were established as 1.42 Mg/m<sup>3</sup> and 31%, respectively. A moisture content of 1.2% OMC of the un-stabilized Kaolinite was adopted in compliance with BS EN 13286-49 [33], with a view of achieving optimum performance for the cylinder specimens developed from the mix compositions. The choice of adopting 1.2% OMC of the un-stabilized Kaolinite was due to the impracticality of obtaining the OMC and MDD values of each mix composition, with a view of achieving optimum performance. Therefore, equal initial weights of the target material were utilized during material quantity calculations, and are expected to be within experimental error, the same volume and bulk density for all the material compositions for each stabilizer dosage and Gypsum content group. Therefore, dry materials capable of producing three (3) compacted cylindrical test specimens, each of dimensions 50 mm in diameter and 100 mm in length, were thoroughly mixed in a mechanical mixer for 2 min before slowly introducing the predetermined amount of water. Intermittent hand mixing with

a palette knife was carried out for another 2 min to achieve a homogeneous mix and ensure that the full potential of stabilization was achieved. Each compacted cylindrical test specimen was made by placing the wet material of each sample in a steel mould fitted with a collar, so as to accommodate all the materials. This material was then subjected to a static compression using a hydraulic jack to achieve the approximated MDD in a loading frame, while the volume was kept constant. The compacted cylinder was left in the mould under pressure for a short period (60 s) before release to allow for relaxation and ultimately permit the dissipation of pore pressure. Thereafter, the cylindrical specimen was carefully thrust out of the steel mould by the application of a steel plunger lubricated with a thin film of oil to ease the removal of the specimen (Figure 1).

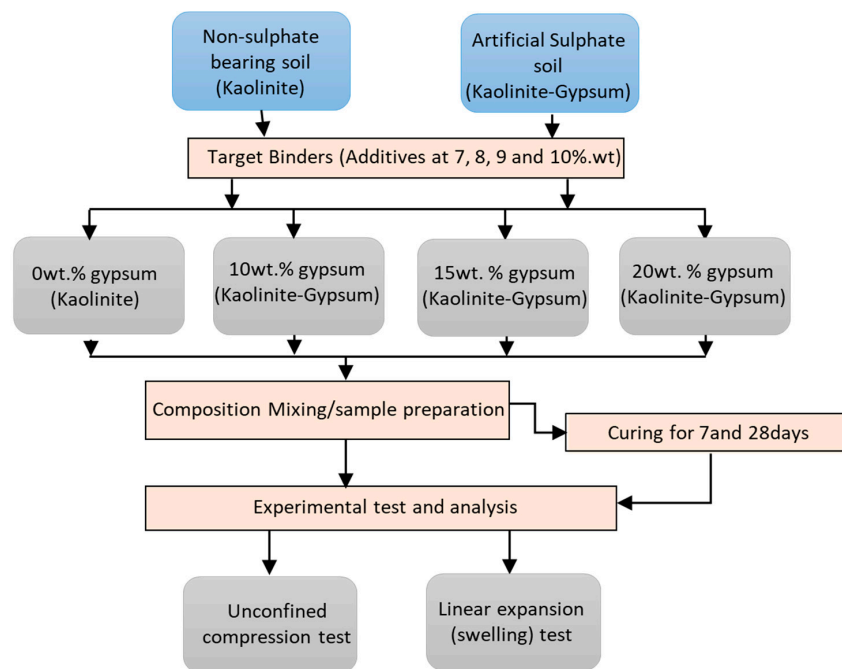
**Table 2.** Control mix design for optimum stabilizer dosage/Gypsum content using CEM I in Kaolinite clay.

Mix Code	Mix Composition	Gypsum Content (%)	Binders (g)			Water (g)	Total Weight (g)
			CEM I	GP	K		
BC0-1	7CEM I:0GP:K	0	18.8	0.0	268.4	106.8	394.0
BC0-2	8CEM I:0GP:K		21.3	0.0	265.9	106.8	394.0
BC0-3	9CEM I:0GP:K		23.7	0.0	263.5	106.8	394.0
BC0-4	10CEM I:0GP:K		26.1	0.0	261.1	106.8	394.0
BC10-1	7CEM I:10GP:K	10	18.8	24.4	244.0	106.8	394.0
BC10-2	8CEM I:10GP:K		21.3	24.2	241.7	106.8	394.0
BC10-3	9CEM I:10GP:K		23.7	24.0	239.5	106.8	394.0
BC10-4	10CEM I:10GP:K		26.1	23.7	237.3	106.8	394.0
BC15-1	7CEM I:15GP:K	15	18.8	35.0	233.4	106.8	394.0
BC15-2	8CEM I:15GP:K		21.3	34.7	231.2	106.8	394.0
BC15-3	9CEM I:15GP:K		23.7	34.4	229.1	106.8	394.0
BC15-4	10CEM I:15GP:K		26.1	34.1	227.0	106.8	394.0
BC20-1	7CEM I:20GP:K	20	18.8	44.7	223.7	106.8	394.0
BC20-2	8CEM I:20GP:K		21.3	44.3	221.6	106.8	394.0
BC20-3	9CEM I:20GP:K		23.7	43.9	219.6	106.8	394.0
BC20-4	10CEM I:20GP:K		26.1	43.5	217.6	106.8	394.0



**Figure 1.** Steel mould with the extruded cylindrical test specimen.

The cylindrical test specimen was then wiped of any loose soil particle or oil stains, weighed, labelled, and wrapped with cling film to ensure minimal loss of moisture. Subsequently, the specimens were placed in a sealed plastic box, stored for moist curing at a temperature of  $20 \pm 2$  °C for a duration of 7, 28, 56 or 90 days prior to testing. The plastic container helped in the regulation of humidity as well as minimization of the deleterious carbonation effect which is common to stabilized soil systems. In total, 176 test cylinder specimens were prepared, 144 of them for testing for strength and 32 for linear expansion (swelling) tests. Figure 2 shows the layout of the experimental procedure.

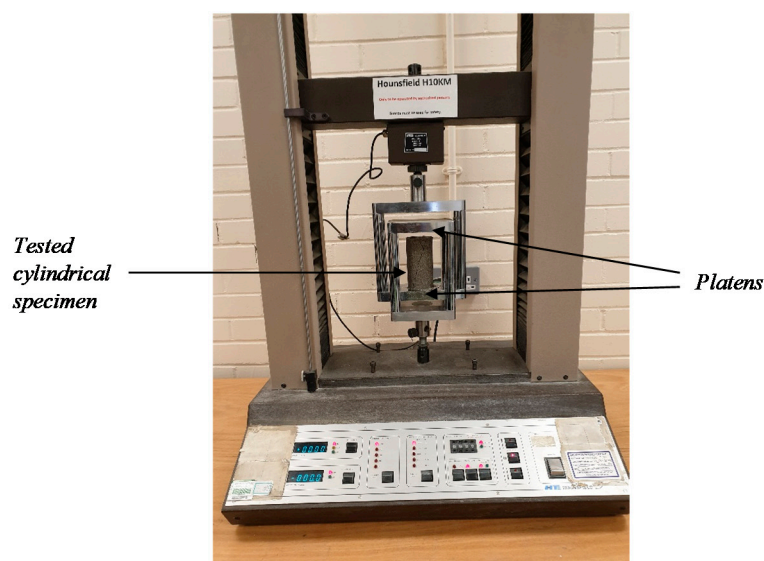


**Figure 2.** Schematic representation of the experimental procedure.

### 2.3. Experimental Test and Analysis

#### 2.3.1. Unconfined Compression Strength (UCS) Test

The UCS test was carried out in accordance with BS 1924 [34] using a Hounsfield Compression Testing machine capable of loading up to 10,000 N. At the end of the curing period, three cylinder test specimens per mix proportion were tested for compressive strength after moist curing ages of 7 and 28 days. The cylindrical test specimen to be tested was placed on the bottom platen of the Hounsfield Compression Testing machine, and the top platen lowered carefully such that the platens very lightly touched both the top and bottom surfaces of the cylindrical test specimen in compliance with BS EN 12390 [35] (Figure 3). A compression strain rate of 1 mm/minute was applied until failure occurred. The maximum load at the point of failure for each cylindrical test specimen was recorded and the mean of three strength values used as the representative UCS value for the mix composition.



**Figure 3.** A Hounsfield Compression Testing machine (10,000 N capacity).

### 2.3.2. Linear Expansion (Swelling) Test

The determination of the swelling/shrinkage performance (%) of the stabilized test specimen was achieved by carrying out a linear expansion test. It was carried out in compliance with BS EN 13286-49 [33] by measuring the amount of expansion. Two cylindrical test specimens representing each of the mix compositions were prepared for resistance to linear expansion upon partial soaking in water after 24 h of sample preparation.

For effective partial soaking, the cling film wrapping the test specimen was carefully trimmed off with a knife to expose about 5mm of the bottom end. Thereafter, a Perspex disc was placed at the top, while a porous disk was placed at the bottom of the test specimen. The test specimen was then lowered onto a Perspex platform within the Perspex cell with the exposed portion of the test specimen placed at the bottom of the Perspex cell (Figure 4). Subsequently, the lid for the Perspex cell which had been fitted with a digital dial gauge to monitor linear expansion was placed as cover, and the digital gauge adjusted to zero. Deionized water was then later introduced into the Perspex cell through the top inlet to about 10mm, so that it covered (soaked) the exposed bottom of the test specimen. The purpose of this soaking was to accelerate the hydration reaction process and establish whether the treatment (stabilization) results into expansion of the stabilized robust material upon soaking [12,19]. Additionally, the use of deionized water was to ensure that the test specimen was not contaminated from any metallic or other ions that could impact on the hydration reaction. It was also important to introduce the water along the sides of the Perspex cell wall, so as not to cause any damage by splashing on the test specimen. The inlet on the plastic container was covered after the introduction of deionized water into the Perspex cell. This was to minimize evaporation, drying out of the test specimens and any available carbon dioxide that would otherwise cause carbonation on the free lime in the hydrating system. The linear expansion measurements were monitored and recorded every 24 h during the partial soaking in water, until no further significant swelling was observed.

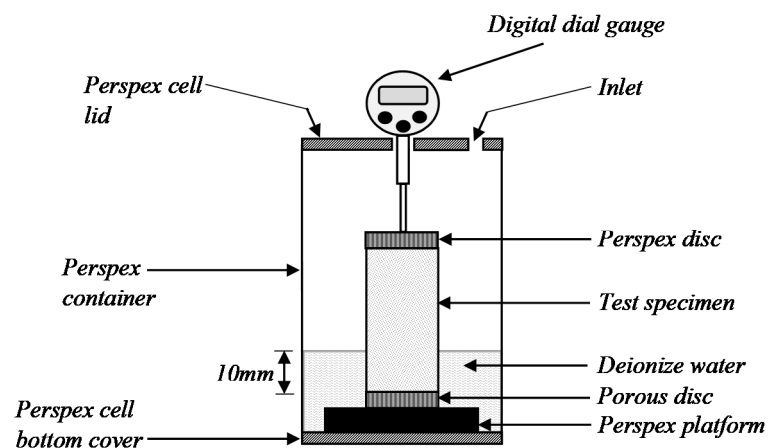


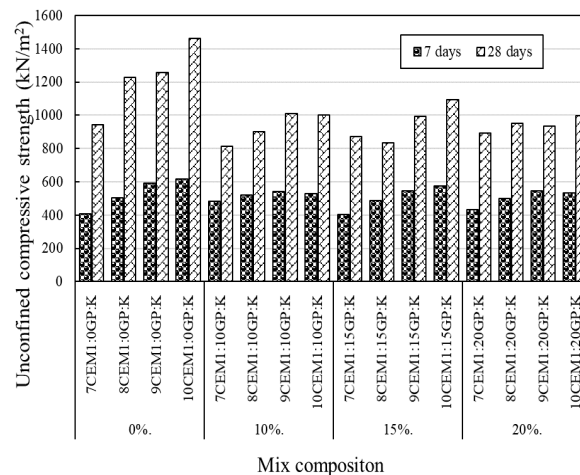
Figure 4. Schematic diagram of a Perspex cell test set-up for monitoring linear expansion.

## 3. Results

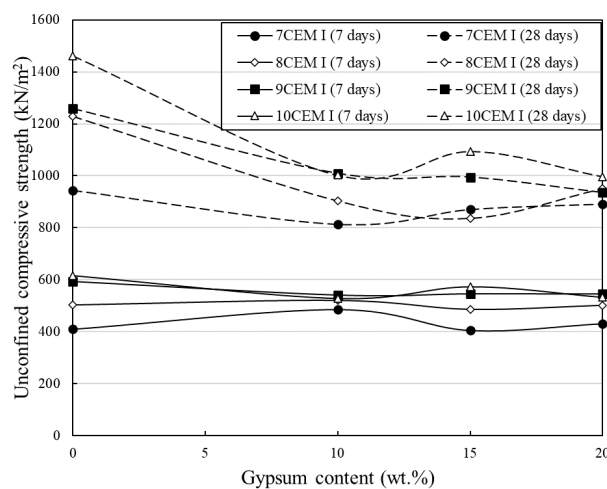
### 3.1. Strength Development of CEM I–Kaolinite and CEM I–Gypsum–Kaolinite Clay Systems

Figure 5 illustrates the Unconfined Compressive Strength (UCS) results for CEM I–Kaolinite clay and CEM I–Kaolinite–Gypsum cylinder test specimens dosed with 0, 10, 15 and 20 wt.% Gypsum content, and stabilized with 7, 8, 9 and 10 wt.% of CEM 1 at both 7 and 28 days moist curing periods. Observations showed that across all the mixes, mixes with the highest CEM I dosage (10 wt.%) produced the highest compressive strength values at 7 and 28 days for both the natural Kaolinite (CEM I–Kaolinite clay), and Kaolinite clay system artificially dosed with sulphate (CEM I–Gypsum–Kaolinite clay systems). The strength was observed to reduce for the CEM I–Gypsum–Kaolinite clay systems when Gypsum was introduced into the stabilized system throughout the curing period, especially at lower cement

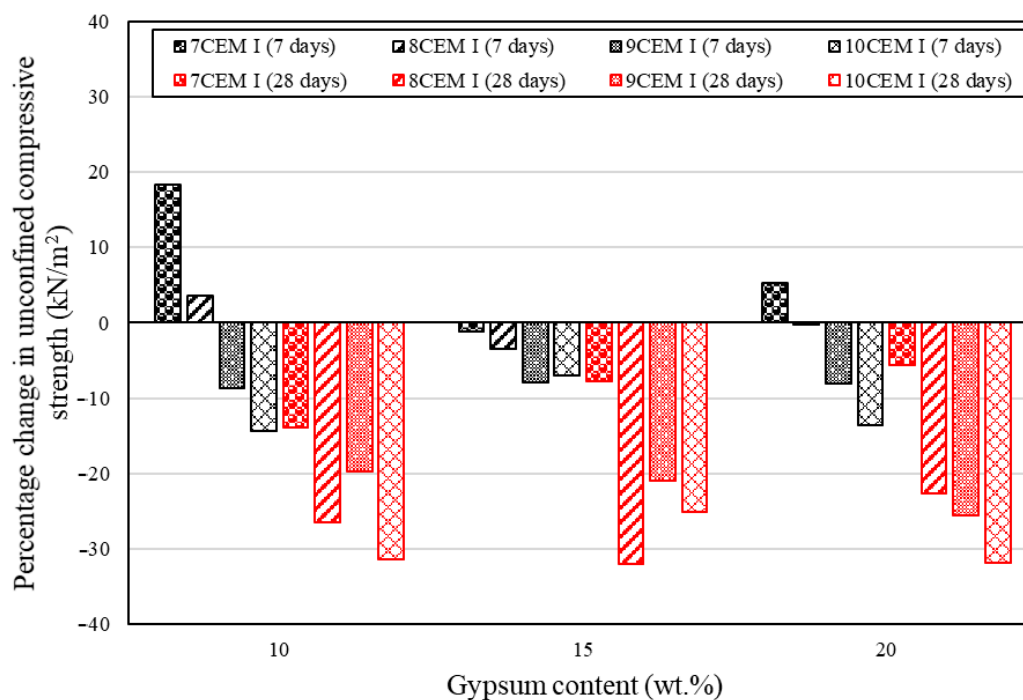
content. The impacts of varying CEM I and Gypsum on the compressive strengths across all the mix compositions at the standardized moist curing period of 28 days can be seen in Figure 6. A small increase in compressive strength values was evident with increasing Gypsum dosage (0–10%) for CEM I–Kaolinite–Gypsum clay cylinder specimens with 7 and 8 wt.% CEM I stabilizer content, while a reduction in strength performance were observed for CEM I–Kaolinite–Gypsum clay cylinder test specimens stabilized with 9 and 10 wt.% CEM I at 7 days curing age. On the contrary, a further increase in Gypsum content (10–20 wt.%) resulted in the reduction in compressive strength values for CEM I–Kaolinite–Gypsum clay systems stabilized with 7 and 8% CEM I, while an increase in compressive strength values was experienced for CEM I–Kaolinite–Gypsum clay systems stabilized with 9 and 10% CEM I at 7 days curing age. The effects of varying Gypsum contents on the unconfined compressive strengths for 7, 8, 9 and 10% CEM I can also be seen in Figure 7 on varying Gypsum content. It showed a significant percentage reduction in compressive strength between the range of 6 to 47% for all the mix compositions at the standardized moist curing period of 28 days. It is not surprising that mix compositions with calcium-based binder content (8 wt.%) produced significant reduction in compressive strength of 36, 47 and 30% at 10, 15 and 20% Gypsum content due to its perceived expansion tendency. This confirms the most deleterious effect of high Gypsum content in a stabilized cylinder specimen using 8 wt.% calcium-based binder.



**Figure 5.** Combined unconfined compressive strength results at 7 and 28 days curing age for Kaolinite clay cylinder samples containing 0, 10, 15 and 20 wt.% Gypsum and stabilized with varying CEM I stabilizer dosages (7, 8, 9 and 10 wt.%).



**Figure 6.** Unconfined compressive strength against Gypsum content for Kaolinite clay–Gypsum–CEM I cylinder samples with varying CEM I and Gypsum content at 7 and 28 days curing age (days).

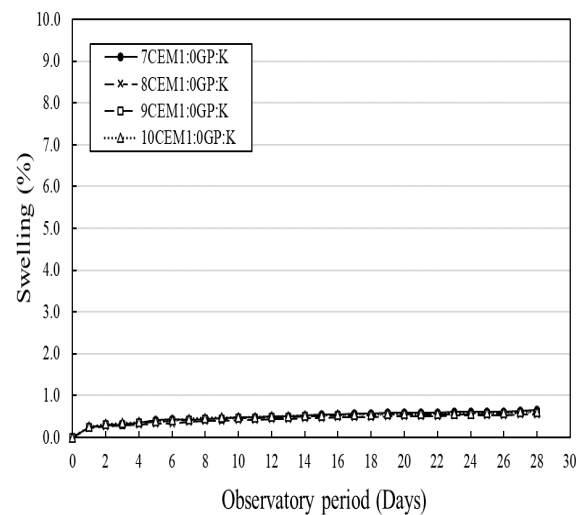


**Figure 7.** Percentage change in unconfined compressive strength against Gypsum content for CEM I–Gypsum–Kaolinite clay cylinder specimen at 7 and 28 days curing age (days).

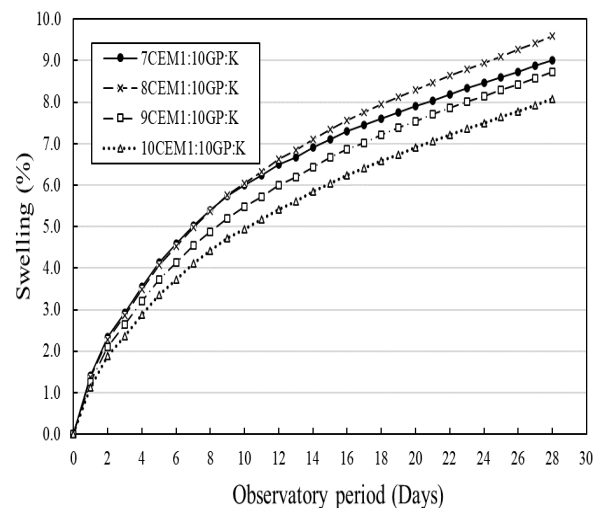
### 3.2. Swell Development of CEM I–Kaolinite and CEM I–Gypsum–Kaolinite Clay Systems

Figures 8–11 illustrate the typical swelling plots for a CEM I–Kaolinite clay and CEM I–Kaolinite–Gypsum clay systems dosed with 0–20 wt.% Gypsum content, and stabilized with 7, 8, 9 and 10 wt.% of CEM I for an observatory period of 28 days. The observed swell was immediate after soaking the cylinder specimen in water, and it was continuous over the observation period of 28 days with no significant shrinkages. The swellings observed were consistent with reported cases of expansion in sulphate bearing soils stabilized with calcium-based materials [16,20,36]. It was evident for the CEM I–Kaolinite systems that the mix with 7 wt.% dosage experienced the maximum expansion of 0.42 and 0.65% at 7 and 28 days curing, respectively, while mix 8CEM I:K produced the lowest swell of 0.37 and 0.55% at 7 and 28 days curing respectively (see Figures 9 and 12). This result showed that lower levels of CEM I acted as good stabilizing agents, which were very effective at reducing swelling in the absence of sulphate (CEM I–Kaolinite systems). However, test results from Figures 9–11 revealed that the introduction of Gypsum (10, 15 and 20 wt.%) modified the behaviour, and properties of the CEM I–Gypsum–Kaolinite clay systems resulted in significant increase in the swelling magnitude. Results also showed that mixes 7CEM I:0GP:K; 8CEM I:10GP:K; 7CEM I:15GP:K and 7CEM I:20GP:K all showed higher magnitudes of swelling after 28 days of observation for all the Kaolinite systems with 0, 10, 15 and 20 wt.% Gypsum content (Figures 9–11). Subsequently, they were further subjected to an increased observation period of 56 days, where they exhibited further increased swelling magnitudes to achieve maximum swellings of 0.65%, 12.6%, 11.2% and 10.1% respectively (Figure 12).

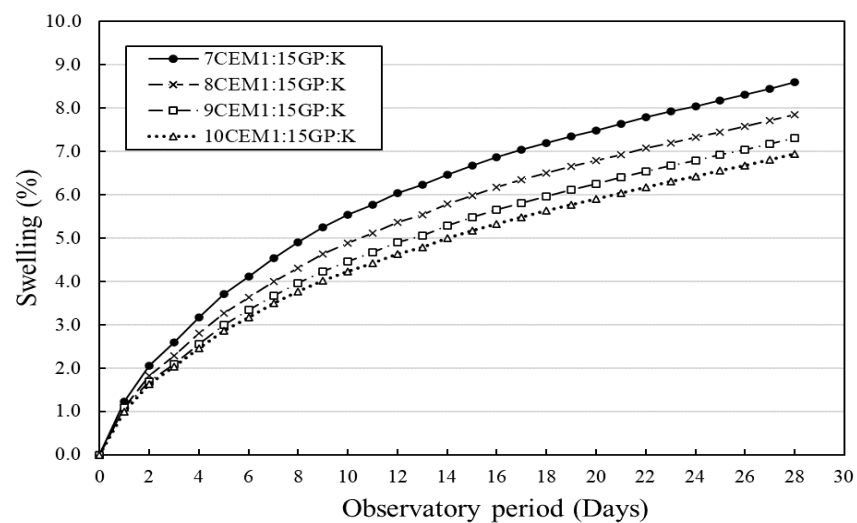




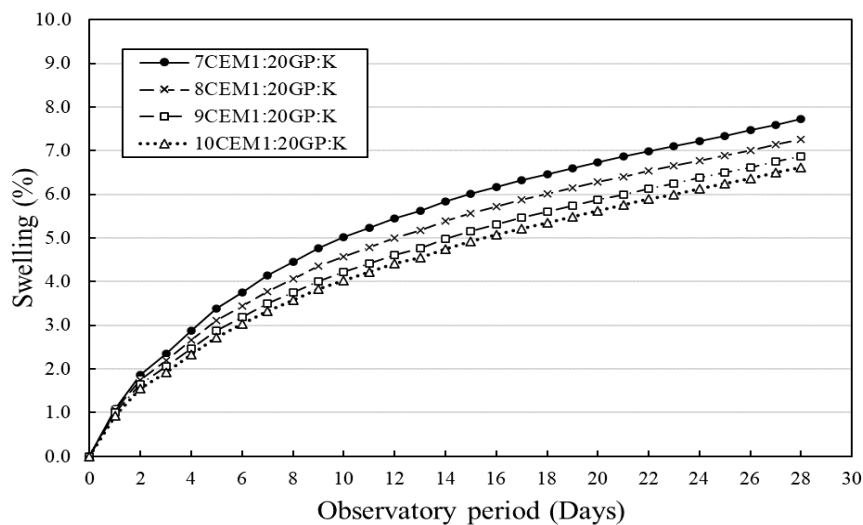
**Figure 8.** Typical plots of swelling against observatory period for CEM I–Gypsum–Kaolinite clay cylinder samples with 0 wt.% Gypsum content stabilized with 7, 8, 9 and 10 wt.% of CEM I.



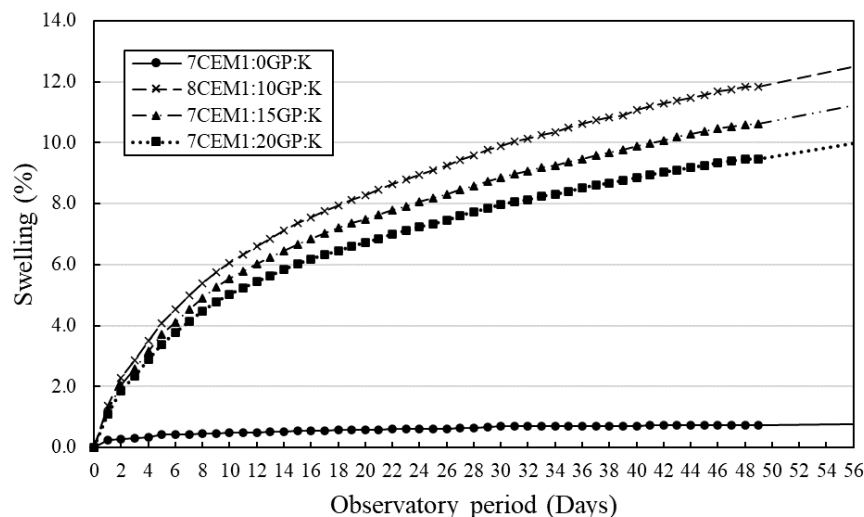
**Figure 9.** Typical plots of swelling against observatory period for CEM I–Gypsum–Kaolinite clay cylinder samples with 10 wt.% Gypsum content stabilized with 7, 8, 9 and 10 wt.% of CEM I.



**Figure 10.** Typical plots of swelling against observatory period for CEM I–Gypsum–Kaolinite clay cylinder samples with 15 wt.% Gypsum content stabilized with 7, 8, 9 and 10 wt.% of CEM I.



**Figure 11.** Typical plots of swelling against observatory period for CEM I–Gypsum–Kaolinite clay cylinder samples with 20 wt.% Gypsum content stabilized with 7, 8, 9 and 10 wt.% of CEM I.



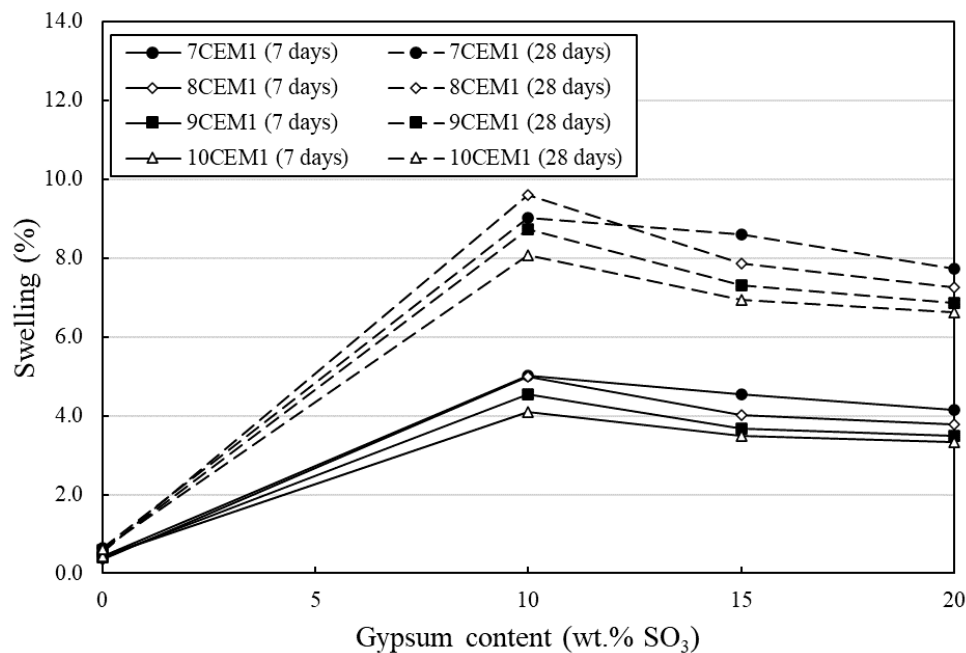
**Figure 12.** Typical swelling plots for maximum swells of Kaolinite clay cylinder samples with varying Gypsum (0–20 wt.%) and CEM I (7 and 8 wt.%) content.

#### 4. Discussion

The development of strength identified across the Kaolinite clay systems that were developed can be attributed to the hydration reactions, which primarily produced the cementitious hydrate—calcium silicate hydrate (C-S-H) gel. This is responsible for the development of strength within the CEM I–Kaolinite clay and CEM I–Kaolinite–Gypsum clay systems. The hydration reactions are complex as they revolve around the modification/strengthening of clay soil particles through ion exchange within the stabilized product. This complex mechanism involves the reaction of calcium oxide (CaO) from CEM I with the silica and alumina present within Kaolinite clay soil particles. Thereafter, an exchange of cation may occur in the whole system, during which sodium ion ( $\text{Na}^+$ ) and potassium ( $\text{K}^+$ ) is displaced. As such, the replacement of monovalent sodium and potassium ions by divalent calcium ion, during the ion exchange process induces changes in the soil properties within the first one hour of the hydration process. Furthermore, the concentration of divalent calcium ions also induces flocculation of the clay particles, which may then transform the plastic soil particles to a granular state, while the monovalent sodium and potassium ions enhance dispersion. [20,37].

The introduction of sulphate increases the available calcium within the artificial sulphate soil system (CEM I–Gypsum–Kaolinite clay system) resulting in increased production of CSH gel. However, it is proposed that the reason for the variation in strength in the stabilized mixtures could be due to the production of increased amount of ettringite compared to the production of CSH gel necessary for strength development. The ettringite crystals are formed in the pores of the soil, which is expected to be filled by the CSH gel to improve on the strength of the stabilized CEM I–Kaolinite–Gypsum clay cylinder test specimens during moist curing [38]. The reduction in compressive strength that was observed after the introduction of 10 wt.% Gypsum may be attributed to the attainment of a critical Gypsum content value, after which any further addition of Gypsum will result in oversaturation of sulphate in the stabilized product. However, the identified increase in compressive strength after the introduction of 15 wt.% Gypsum for the cylinder samples could be due to the increase in the formation of more ettringite crystals, which can have a positive impact on the compressive strength [38]. The increase in strength is more evident at 28 days moist curing age due to the constant increase in the hydration reaction within the CEM I–Gypsum–Kaolinite clay system. The significant increase in linear expansion for CEM I stabilized soils with different Gypsum levels could also be attributed to the formation of ettringite  $[\text{Ca}_6\text{Al}_2(\text{SO}_4)_3(\text{OH})_{12}\cdot 26\text{H}_2\text{O}]$  obtained from the hydration reaction of calcium (obtained from CEM I), alumina, silica, sulphate in the presence of water [20–23]. Ettringite develops on the surface of the clay particles during the curing process. However, in saturated conditions (such as the soaking regime), ettringite has the capability of causing a rapid increase in volume due to the absorption of large amounts of water. This resulted in a dramatic increase in swelling for the simulated Kaolinite clay–Gypsum system, which utilizes CEM I as a calcium-based stabilizer compared with a non-sulphate Kaolinite clay system [15]. Another possible explanation for the increased expansion of CEM I–Kaolinite–Gypsum clay systems compared with CEM I–Kaolinite systems could perhaps be the hydration of Gypsum used for the simulation of the artificial sulphate soil system. This could lead to swelling by filling up of voids within the stabilized system with water due to the expansion of the Gypsum crystals (Gypsum crystallization). However, the occurrence and formation of Gypsum crystals could be in different forms. The most prominent type are the prismatic crystals which are formed in a clustered disc shaped rosettes, or a thin acicular crystal that exerts pressure on the CEM I–Kaolinite–Gypsum clay systems to cause expansion as the Gypsum crystals develop in size [38–41].

The comparative impact of varying Gypsum content with the recorded swellings for the artificial sulphate soil systems (Figure 13) indicated a rapid increase in swelling shortly after soaking the cylindrical test specimens. This was evident for mixes stabilized with 7, 8, 9 and 10 wt.% calcium content and dosed with Gypsum content of 0–10 wt.%, which later decreased with successive increases in Gypsum content (10–20 wt.%). This could be due to the slower hydration reaction experienced using large amounts of Gypsum content (10–20 wt.%), which oversaturated the artificial sulphate soil systems compared with a faster hydration reaction experienced for low Gypsum content (0–10 wt.%) at both 7 and 28 days curing period. However, the observed reduction in CEM I content at both 7 and 28 days of observation produced increased swelling magnitudes for every percentage increase in Gypsum content. This gives an indication that a specific CEM I (calcium) content is required in relation to other components/reactants to reduce oversaturation of calcium within the CEM I–Gypsum–Kaolinite clay system for optimum swelling. According to Wang et al. [12], there are various severity levels with respect to expansion as seen in Table 3. This shows a “very strong” severity level that relates to the maximum swell of 9.6% obtained in the current research for mix 8CEM I:10GP:K.



**Figure 13.** Swelling against Gypsum content for CEM I–Gypsum–Kaolinite clay cylinder samples with varying CEM 1 and Gypsum content at 7 and 28 days curing ages (days).

**Table 3.** Severity levels for swelling in clay soils.

Swelling (%)	Severity
0	No swell
0–0.1	Negligible
0.1–0.5	Light
0.5–1.0	Medium
1.0–2.0	Strong
Over 2.0	Very strong

## 5. Conclusions

The ability to simulate an artificial sulphate soil system with high dosages of Gypsum was quite expository, as it aided the understanding of the mechanism and establishment of binder content limits for expansion when stabilized with a calcium-based material (CEM I). The outcomes from this study showed the impacts of sulphate on the swell and strength properties of an artificial sulphate soil system. The following conclusions can be drawn:

1. The maximum expansion for an artificial sulphate soil system occurred at 8 wt.% binder content with an optimum Gypsum content of 10% by weight.
2. The swell behaviour of Kaolinite clay–Gypsum system stabilized with calcium-based product is dependent on both the Gypsum content and curing age.
3. The swelling experienced in the artificial sulphate soil system stabilized with calcium-based products-CEM I was mainly due to the formation of ettringite and Gypsum crystallisation in the presence of water.
4. The increase in strength for the cylinder samples are largely due to the continuous hydration reaction, which produces more C-S-H gel to fill the voids within the sulfate induced Kaolinite soil particles.
5. The introduction of Gypsum to a Kaolinite clay system reduces the compressive strength of the stabilised product with a range factor of 6 to 47% at 28 days moist curing age.

**Author Contributions:** Conceptualization, B.A., J.K. and J.O.; methodology, B.A.; validation, B.A., J.K. and J.O.; formal analysis, B.A.; investigation, B.A.; writing—original draft preparation, B.A.; writing—review and editing, J.K. and J.O.; visualization, B.A.; supervision, J.K. and J.O.; project administration, B.A. All authors have read and agreed to the published version of the manuscript.

**Funding:** This research received no external funding.

**Conflicts of Interest:** The authors declare no conflict of interest.

## References

1. Jan, P.; Šedivý, M. Prediction of Soil Swelling Parameters. *Procedia Earth Planet. Sci.* **2015**, *15*, 219–224. [[CrossRef](#)]
2. Dang, L.C.; Fatahi, B.; Khabbaz, H. Behaviour of expansive clay stabilized with hydrated lime and Bagasse Fibres. *Procedia Eng.* **2016**, *143*, 658–665. [[CrossRef](#)]
3. Jones, L.D.; Jefferson, I. Expansive soils. In *ICE Manual of Geotechnical Engineering: Volume 1, Geotechnical Engineering Principles, Problematic Soils and Site Investigation*; Burland, J., Ed.; ICE Publishing: London, UK, 2012; pp. 413–441.
4. Kinuthia, J.M.; Oti, J.E. Designed non-fired clay mixes for sustainable and low carbon use. *Appl. Clay Sci.* **2012**, *59*, 131–139. [[CrossRef](#)]
5. Miqueleiz, L.; Ramírez, F.; Seco, A.D.A.; Nidzam, R.; Kinuthia, J.; Abu Tair, A.; Garcia, R. The use of stabilised Spanish clay soil for sustainable construction materials. *Eng. Geol.* **2012**, *133–134*, 9–15. [[CrossRef](#)]
6. Phanikumar, B.R.; Singla, R. Swell-consolidation characteristics of fibre-reinforced expansive soils. *Soils Found.* **2016**, *56*, 138–143. [[CrossRef](#)]
7. Cheshomi, A.; Eshaghi, A.; Hassanpour, J. Effect of lime and fly ash on swelling percentage and Atterberg limits of sulfate-bearing clay. *Appl. Clay Sci.* **2017**, *135*, 190–198. [[CrossRef](#)]
8. Seco, A.; Miqueleiz, L.; Prieto, E.; Marcelino, S.; García, B.; Urmeneta, P. Sulfate soils stabilization with magnesium-based binders. *Appl. Clay Sci.* **2017**, *135*, 457–464. [[CrossRef](#)]
9. Seco, A.; Ramirez, F.; Miqueleiz, L.; Garcia, B. Stabilization of expansive soils for use in construction. *Appl. Clay Sci.* **2011**, *51*, 348–352. [[CrossRef](#)]
10. Cheng, K.; Heidari, Z. A new method for quantifying cation exchange capacity in clay minerals. *Appl. Clay Sci.* **2018**, *161*, 444–455. [[CrossRef](#)]
11. Schanz, T.; Tripathy, S.; Sridharan, A. Volume change behaviour of swelling and non-swelling clays upon inundation with water and a low dielectric constant fluid. *Appl. Clay Sci.* **2018**, *158*, 219–225. [[CrossRef](#)]
12. Wang, L.; Roy, A.; Seals, R.K.; Metcalf, J.B. Stabilization of Sulfate-Containing Soil by Cementitious Mixtures Mechanical Properties. *Transp. Res. Rec. J. Transp. Res. Board* **2003**, *1837*, 12–19. [[CrossRef](#)]
13. Oti, J.E.; Kinuthia, J.; Bai, J. Compressive strength and microstructural analysis of unfired clay masonry bricks. *Eng. Geol.* **2009**, *109*, 230–240. [[CrossRef](#)]
14. Oti, J.E.; Kinuthia, J.; Bai, J. Engineering properties of unfired clay masonry bricks. *Eng. Geol.* **2009**, *107*, 130–139. [[CrossRef](#)]
15. Kinuthia, J.; Wild, S.B.; Jones, G. Effects of monovalent and divalent metal sulphates on consistency and compaction of lime-stabilised kaolinite. *Appl. Clay Sci.* **1999**, *14*, 27–45. [[CrossRef](#)]
16. Kinuthia, J.M.; Wild, S. Effects of some metal sulphates on the strength and swelling properties of lime-stabilised kaolinite. *Int. J. Pavement Eng.* **2001**, *2*, 103–120.
17. Rahmat, M.N.; Kinuthia, J.M. Effects of mellowing sulfate-bearing clay soil stabilized with wastepaper sludge ash for road construction. *Eng. Geol.* **2011**, *117*, 170–179. [[CrossRef](#)]
18. Nidzam, R.M.; Kinuthia, J.M. Sustainable soil stabilisation with blastfurnace slag—A review. *Proc. Inst. Civ. Eng. Constr. Mater.* **2010**, *163*, 157–165. [[CrossRef](#)]
19. Caselles, L.D.; Hot, J.; Roosz, C.; Cyr, M. Stabilization of soils containing sulfates by using alternative hydraulic binders. *Appl. Geochem.* **2020**, *113*, 104494. [[CrossRef](#)]
20. Wild, S.; Kinuthia, J.; Jones, G.; Higgins, D. Suppression of swelling associated with ettringite formation in lime stabilized sulphate bearing clay soils by partial substitution of lime with ground granulated blastfurnace slag (GGBS). *Eng. Geol.* **1999**, *51*, 257–277. [[CrossRef](#)]
21. Ciliberto, E.; Ioppolo, S.; Manuella, F. Ettringite and thaumasite: A chemical route for their removal from cementitious artefacts. *J. Cult. Herit.* **2008**, *9*, 30–37. [[CrossRef](#)]

22. Norman, R.L.; Dann, S.E.; Hogg, S.C.; Kirk, C.A. Synthesis and structural characterisation of new ettringite and thaumasite type phases:  $\text{Ca}_6[\text{Ga}(\text{OH})_6 \cdot 12\text{H}_2\text{O}]_2(\text{SO}_4)_3 \cdot 2\text{H}_2\text{O}$  and  $\text{Ca}_6[\text{M}(\text{OH})_6 \cdot 12\text{H}_2\text{O}]_2(\text{SO}_4)_2(\text{CO}_3)_2$ ,  $\text{M} = \text{Mn}, \text{Sn}$ . *Solid State Sci.* **2013**, *25*, 110–117. [[CrossRef](#)]
23. Hunter, D. Lime-induced heave in sulfate-bearing clay soils. *J. Geotech. Eng.* **1988**, *114*, 150–167. [[CrossRef](#)]
24. Dermatas, D. Ettringite-Induced Swelling in Soils: State-of-the-Art. *Appl. Mech. Rev.* **1995**, *48*, 659–673. [[CrossRef](#)]
25. Snedker, E.A.; Temporal, J. M40 Motorway Banbury IV Contract—Lime Stabilization. *Highw. Transp.* **1990**, *37*, 7–8.
26. Parker, D. 25% of A10 Hit by Sulphide Attack. 2004. Available online: <https://www.newcivilengineer.com/archive/25-of-a10-hit-by-sulphide-attack-25-03-2004> (accessed on 20 July 2020).
27. Li, W.; Yi, Y.; Puppala, A.J. Suppressing Ettringite-Induced Swelling of Gypseous Soil by Using Magnesia-Activated Ground Granulated Blast-Furnace Slag. *J. Geotech. Geoenviron. Eng.* **2020**, *146*, 6020008. [[CrossRef](#)]
28. BS EN 197-1:2011: *Cement Part 1: Composition, Specifications and Conformity Criteria for Common Cements*; BSI Standards Ltd.: London, UK, 2011.
29. BS EN 15167-1:2006: *Ground Granulated Blast Furnace Slag for Use in Concrete, Mortar and Grout—Definitions, Specifications and Conformity Criteria*; BSI Standards Ltd.: London, UK, 2006.
30. BS EN 15309:2007: *Characterization of Waste and Soil—Determination of Elemental Composition by X-Ray Fluorescence*; BSI Standards Ltd.: London, UK, 2007.
31. BS ISO 18227:2014: *Soil Quality, Determination of Elemental Composition by X-Ray Fluorescence*; BSI Standards Ltd.: London, UK, 2014.
32. BS 1377-4:1990: *Methods of Test for Soils for Civil Engineering Purposes: Compaction-Related Tests*; BSI Standards Ltd.: London, UK, 1990.
33. BS EN 13286-49:2004, *Unbound and Hydraulically Bound Mixtures: Accelerated Swelling Test for Soil Treated by Lime and/or Hydraulic Binder*; BSI Standards Ltd.: London, UK, 2004.
34. BS 1924-2:2018, *Hydraulically Bound and Stabilized Materials for Civil Engineering Purposes: Sample Preparation and Testing of Materials during and after Treatment*; BSI Standards Ltd.: London, UK, 2018.
35. BS EN 12390-4:2019: *Testing Hardened Concrete—Part 4: Compressive Strength—Specification for Testing Machines*; BSI Standards Ltd.: London, UK, 2019.
36. Behnood, A. Soil and clay stabilization with calcium- and non-calcium-based additives: A state-of-the-art review of challenges, approaches and techniques. *Transp. Geotech.* **2018**, *17*, 14–32. [[CrossRef](#)]
37. Ikeagwuani, C.C.; Nwonu, D.C. Emerging trends in expansive soil stabilisation: A review. *J. Rock Mech. Geotech. Eng.* **2019**, *11*, 423–440. [[CrossRef](#)]
38. Kuttah, D.K.; Sato, K. Review on the effect of gypsum content on soil behavior. *Transp. Geotech.* **2015**, *4*, 28–37. [[CrossRef](#)]
39. Hawkins, A.B.; Pinches, G.M. Understanding sulphate generated heave resulting from pyrite degradation. In *Implications of Ground Chemistry and Microbiology for Construction*; Hawkins, A.B., Ed.; Balkema: Rotterdam, The Netherland, 1997.
40. Abbey, S.J.; Eyo, E.; Oti, J.E.; Amakye, S.Y.; Ng’Ambi, S. Mechanical Properties and Microstructure of Fibre-Reinforced Clay Blended with By-Product Cementitious Materials. *Geosciences* **2020**, *10*, 241. [[CrossRef](#)]
41. Oti, J.; Kinuthia, J. The Development of Stabilised Clay-Hemp Building Material for Sustainability and Low Carbon Use. *J. Civ. Eng. Constr.* **2020**, *9*, 205–214. [[CrossRef](#)]

**Publisher’s Note:** MDPI stays neutral with regard to jurisdictional claims in published maps and institutional affiliations.



© 2020 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (<http://creativecommons.org/licenses/by/4.0/>).