



Article

Experimental Study on Early Age Characteristics of Lime-GGBS-Treated Gypseous Clays under Wet–Dry Cycles

Samuel J. Abbey ^{1,*}, Eyo U. Eyo ¹ and Jeremiah J. Jeremiah ²

¹ Faculty of Environment and Technology, University of the West of England, Bristol BS16 1QY, UK; eyo.eyo@uwe.ac.uk

² Candidate in Civil Engineering Cluster, Department of Geography and Environmental Management, Faculty of Environment and Technology, University of the West of England, Bristol BS16 1QY, UK; jeremiah.jeremiah@uwe.ac.uk

* Correspondence: samuel.abbey@uwe.ac.uk

Abstract: Gypseous soils are capable of presenting ground construction challenges to civil and geotechnical engineers due to their unpredictable deformation characteristics. These undesirable responses are sometimes caused by environmental changes in moisture content due to temperature variations, fluctuation of underground water table, surface water, and gypsum content. Hence, the adoption of effective and economical means of stabilising gypseous soils is imperative. This study's focus is on the early age strength and microstructural characteristics of gypseous soils treated with lime and GGBS. Treated and untreated gypseous soils with 5%, 15%, and 25% gypsum content were subjected to wet–dry cycles while investigating unconfined compressive strength (UCS), water absorption, pH, microstructural changes, and swell. The analysis of the results shows that at zero cycle, the UCS of the untreated gypseous soils increases from 0.62 to 0.79 MPa and swell decreases from 69 to 23%, respectively, as gypsum content increases. However, upon subjection to wet–dry cycles, the UCS reduced from 0.16 to 0.08 MPa at the end of the sixth cycle due to dissolution of gypsum within the soil pores which reduced the strength. The result also shows that gypsum content increases water absorption and reduces the pH of the untreated gypseous soils because of the neutral pH of gypsum. Furthermore, lime-GGBS-treated gypseous soils maintained a higher pH after six wet–dry cycles compared to untreated gypseous soils due to the high pH of lime and the increase in calcium content which improved bonding. In addition, microstructural analysis using SEM indicated early age precipitation of cementitious compounds (CSH) for increasing strength of lime-GGBS-treated gypseous soils compared to untreated gypseous soils.

Keywords: lime-GGBS; gypseous soil; cyclic UCS; wet–dry cycles; soil treatment



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1. Introduction

Calcium-based additives such as cement, and lime have been widely used in improving many problematic ground conditions the world over. Conditions such as swelling due to expansive soil behaviours and low shear strengths of weak clays have been successfully improved to meet engineering requirements by the addition of cement and lime as reported in the literature [1–10]. However, gypseous soils have been reported as presenting unique difficulties in engineering applications due to their susceptibility to collapse during water ingress. The fine and lightweight characteristics of the soil particles and low frictional resistance of unsaturated gypseous soils are worsened in the presence of water, leading to sudden instability [11]. In arid regions where these soils are predominant, losses resulting from the failures of such soils are reported in billions of dollars. Gypseous soils can be classified based on gypsum content according to Table 1.

Table 1. Gypseous soil classification [12].

Gypsum Content (%)	Classification
0–0.3	Non-gypseous
0.3–3	Very lightly gypseous
3–10	Slightly gypseous
10–25	Moderately gypseous
25–50	Highly gypseous

To mitigate the collapsibility of gypseous soils, several additives have been studied. However, the traditional stabilisation methods using ordinary Portland cement (OPC) and lime alone have not been completely satisfactory. In OPC-treated gypseous soils, the formation of hydrated calcium aluminate sulphate (ettringite) $\text{Ca}_6\text{Al}_2(\text{SO}_4)_3(\text{OH})_{12}\cdot 26\text{H}_2\text{O}$ within interparticle surfaces causes swelling and subsequent cracking of the treated soils with adverse effects on the overlying facilities [13–21]. It is therefore desirable to mitigate this effect by using binder combinations which would limit the supply of calcium hydroxide, the major driver of the formation of ettringite. The use of more silica-alumina-based treatment such as alkaline activation of silica-alumina precursors is perhaps one method of improving the interparticle bonds and reducing swell potential of treated gypseous soils. The study by [22] investigated the application of carbide slag-activated GGBS on the engineering characteristics of gypseous soil, and found that the initial formation of ettringite and synthesis of geopolymeric chains reduced the $\text{Ca}(\text{OH})_2$ concentration. This resulted to a reduction in further formation of ettringite development within the matrix of the treated gypseous soils thereby leading to a decrease in swell potential when compared to treatment with OPC. The findings align with previous works by [23–34]. Furthermore, in line with improving swelling of gypseous soils, the experimental study by [25] employed several additives in the treatment of high sulphate-bearing soils including polymers, pulverised fuel ash (PFA), enzymes, and acid salts and reported that the combination of lime and GGBS proved more effective and economical in reducing swell and improving the strength of the treated samples. Additionally, [26] used a combination of lime and PFA to improve the mechanical properties of gypseous soils. The lime was used to compensate for the low calcium oxide (CaO) content of the PFA for adequate formation of strength giving compounds such as calcium silicate hydrates (CSH) and calcium aluminate hydrates (CAH). In another study, acrylate polymer liquid was utilised to improve the compressibility of treated gypseous soil due to increased cohesion caused by coating of soil particles by the acrylate liquid [11]. The addition of ammonium sulphates $(\text{NH}_4)_2\text{SO}_4$ and potassium chlorides (KCl) has also been reported to improve the compressibility and collapse potential of gypseous soils [27]. The method of grouting has been utilised in stabilising different problematic soils including loose materials such as gypseous soils. The isolation and waterproofing effects of grout on gypseous soils was highlighted by [28,29] and is an advantage as protection of the solid grains significantly alleviates the rate of leaching of gypsum within the soil and improves the interparticle bonds. The study by [30] also explored nanotechnology based materials as additive in enhancing the performance of stabilised clays. In the United Kingdom, the combination of GGBS and lime have also been reported to yield improvements in strength while reducing the swell potential of lime-GGBS-treated sulphate soils [13,31–33]. Cement blended with up to 80% GGBS and 10% lime is known to inhibit sulphate-induced heave even in the presence of ettringite [34,35]. This resistance appears to be the result of modifications of the morphology of ettringite when GGBS was used as reported by [36]. Other field applications of GGBS in stabilising sulphate-bearing soils in the UK as reported by [35] include the Baddersley Colliery stabilisation which utilised GGBS and OPC in stabilising 140,000 m² of sulphate and sulphide-bearing clays, Silverstone Racetrack stabilisation which utilised GGBS and quick lime in stabilising 55,000 m² of sulphate-bearing clays, stabilisation of 10,000 m² of sulphate-bearing clay using GGBS and lime at Hud-

dersfield to mention a few. It is also important to recall that an additional concern for stabilised gypseous soils is the formation of ettringite in cement and lime stabilised soils. Additionally, in road subgrade and subbase applications, it is imperative to understand the early-age performance of treated gypseous soil. In the present study, the effects of lime and GGBS treatment on the early age characteristics of moderately gypseous clays subjected to cyclic wetting and drying is investigated. The early age wet and dry cycles of lime and GGBS-treated gypseous soils was studied in other to evaluate the performance of treated gypseous soils under changing environmental conditions characterised by changing moisture contents.

2. Materials and Methods

The materials used in this study consisted of Kaolin clay, bentonite, lime, and ground granulated blast furnace slag (GGBS). In the first instance, artificially synthesised soil (Soil 1), consisting of 50% kaolin clay and 50% bentonite was mixed thoroughly for about 15–20 min in a laboratory soil mixer, and by hand-mixing to ensure a homogenous composition. The grain size distribution of selected materials and the oxide compositions are presented in Figure 1a and Table 2, respectively.

To conduct a precise laboratory study, representative samples of soil 1 were mixed with 5%, 15% and 25% of gypsum by weight of dry soil, and hereafter referred to as gypseous soils (soil 2, soil 3 and soil 4) with varying gypsum contents. The gypseous soils were treated with a combination of 4% lime and 8% GGBS, and the amount of water added to soil–gypsum–binder system was determined based on the optimum moisture content of the soil–gypsum–binder system with an additional amount of water to accommodate for the binders used based on research and experience [31]. The 4% lime used has been previously obtained as the optimum lime content (OLC) from author’s previous work following the procedure outlined in [37], see Figure 1b. The OLC has been defined as the minimum lime content required to cause reduction in plasticity and swell potential of a reactive soil, and for the initiation of mid- to long-term pozzolanic reactions [35,38].

Further thorough mixing of the materials with water was carried out for about 10–15 min to prevent the development of ‘hot spots’ and non-uniformly migrated ions as well as improving the chances of uniform ettringite nucleation site distribution [18]. Samples of the treated gypseous soils were cured in open air for 7 days to allow for sufficient early age strength development. Thereafter, representative treated samples were subjected to wet–dry cycles for up to 6-cycles. During the wet–dry cycles, samples were soaked in water for up to 48 h and oven dried for 24 h at 60 °C before testing.

Table 2. Chemical composition of soil and binders.

Oxide	Kaolinite (%)	Bentonite (%)	Gypsum (%)	Lime (%)	GGBS (%)
SiO ₂	49	57.1	10.93	1	34.1
Al ₂ O ₃	36	17.79	2.88	0.3	13
Fe ₂ O ₃	0.75	4.64	1.16	0.5	0.51
CaO	0.06	3.98	26.32	94	39
MgO	0.3	3.68	-	2	9.5
K ₂ O	1.85	0.9	0.83	-	0.5
TiO ₂	0.02	0.77	0.15	-	1.3
Na ₂ O	0.1	3.27	0.18	-	0.3
SO ₃	-	0.11	34.70	-	0.3
Mn ₂ O ₃	-	0.06	-	1.2	0.7
LOI	12	7.85	19.80	-	1.9

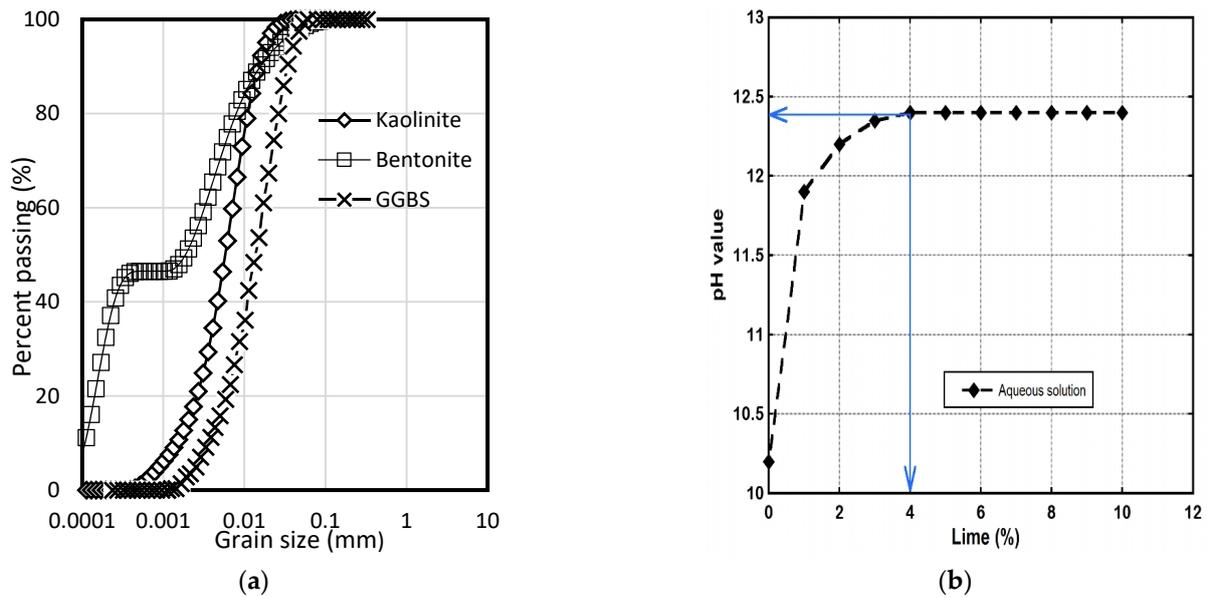


Figure 1. (a,b). Analysis of material grain size and determination of OLC: (a) material grain size; (b) optimum lime content.

Experimental Testing

Prior to the unconfined compressive strength test, samples were compacted in a 105 mm diameter by 115 mm height steel mould, extruded and cured under water for 7 days. A total of 112 samples were prepared for UCS while 8 samples were prepared for swell test making a total of 120 samples utilised for the experimental programme. The treated gypseous soils were tested for UCS, expansion, pH after each cycle of wetting and drying period, as shown in Figure 2. The UCS test was conducted in accordance with [39] on the compacted treated gypseous soils at the end of each wet–dry cycle. To account for any possible swell on the compacted treated gypseous samples, the free swell oedometer test was carried out on the cured samples in line with [40]. Image analysis using the scanning electron micrograph (SEM) was conducted on treated soil with 5% gypsum content to provide a description of the mechanism of change in the fabric of the samples of natural and treated gypseous soils. Small-sized chunks derived from selected originally compacted and cured samples were used to obtain the micrographs. 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, and thereafter, the micrographs (SEM) were collected using the ZEISS EVO equipment.

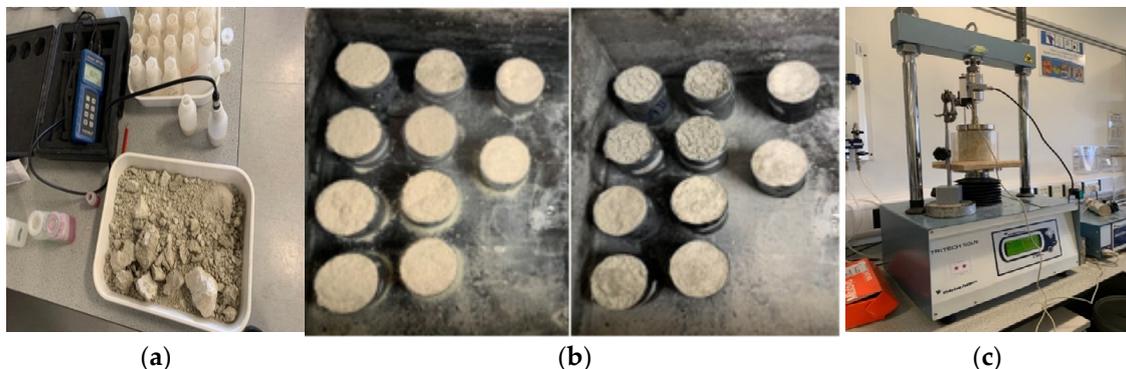


Figure 2. (a–c). Laboratory tests on samples of gypseous soil: (a) laboratory pH testing; (b) selected samples after wet–dry cycle; (c) sample during UCS test.

3. Discussion of Results

3.1. Cyclic UCS

The cyclic UCS refers to the unconfined compressive strength of the untreated and treated gypseous soils under wetting and drying cycles. The results plotted in Figure 3 show the cyclic UCS of both the untreated and treated gypseous soils under the influence of wet–dry cycles. Firstly, it was observed that the initial UCS of the untreated gypseous soils at zero cycles, increased with increasing gypsum content. This increase in UCS followed increase in gypsum content of the gypseous soils. The initial increase in the UCS with increasing gypsum content can be attributed to more calcium availability within the matrix of the gypseous soil as gypsum content increases in the presence of water thereby leading to early age strength development [41–43]. However, upon subjecting the untreated samples to repeated cycles of wetting and drying, the strength of the untreated gypseous soils decreased from 0.79 to 0.08 MPa in order of increasing gypsum content at the end of the sixth cycle. The reduction in UCS was due to the development of desiccation cracks initiated attributed to the dissolution of gypsum from the pores of the gypseous soil under repeated wet–dry cycles which subsequently leads to increase in the volume shrinkage of the untreated soils with further cycles of wetting and drying [44]. It was also observed that the untreated soils with higher gypsum content experienced more profound reduction in UCS than untreated soils with lower gypseous content. This result aligns with the findings of [34,45–47].

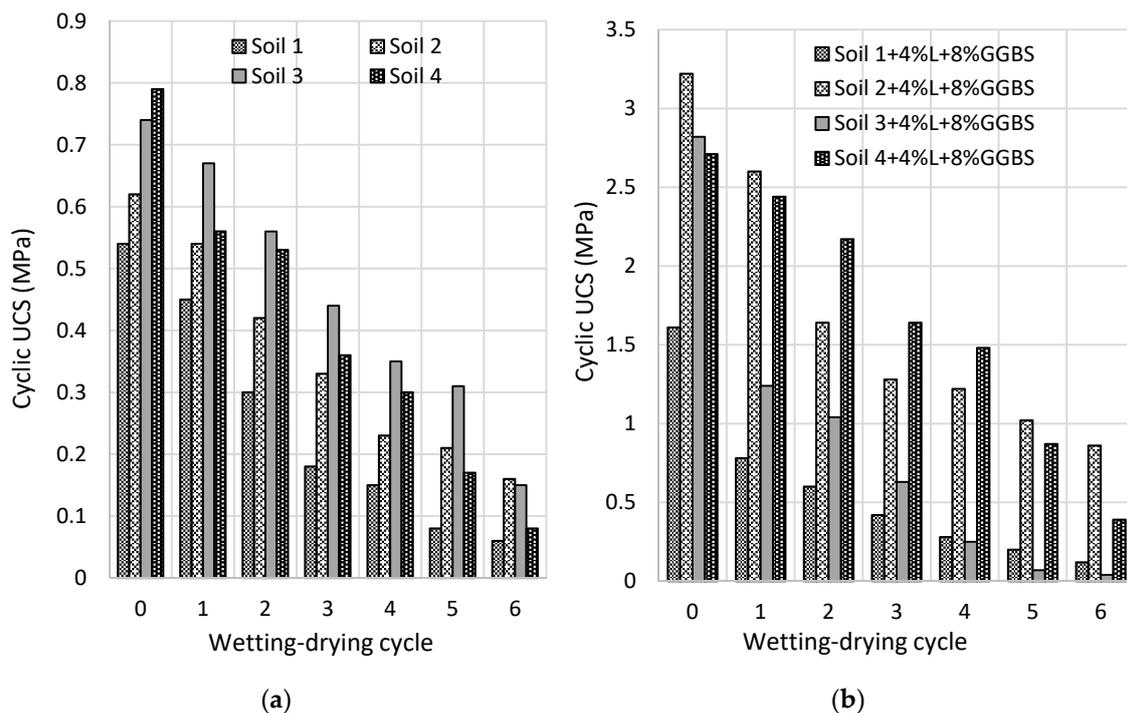


Figure 3. Cyclic UCS and wetting–drying cycles. (a) Untreated gypseous soil, (b) Treated gypseous soil.

Therefore, it can be said that the gypseous soils with lower gypsum content performed better at the end of the 6th cycle, as shown in Figure 3. On the other hand, the UCS of the treated gypseous soils increased with the addition of 4% lime and 8% GGBS. The increase in strength of the treated soil is due to stronger bonding of soil particles due to more available calcium, alumina, and silica contents for faster development of bonds within the matrix of the treated soil. As shown in Table 2 above, the GGBS utilised is characterised by 39% and 34% calcium oxide and silicate content, respectively. This relatively high availability of CSH improves the strength of stabilised clays. More so, the addition of lime reduced the diffused double layer of water within the lime–GGBS–treated gypseous soil matrix which also improved the strength at the end of the 6th cycle. The increase in UCS from

the lime and GGBS treatment was as expected and agrees with other studies by [35,36]. Comparing the strength of the treated and untreated gypseous soils it is obvious that the addition of 4% lime and 8% GGBS improved the UCS of the treated samples by 431%. This, therefore, implies higher resistance of the treated lime-GGBS gypseous soils to the effects of shrinkage-induced cracking under wet–dry cycles as evidenced on the dense particle packing and reduced voids at the microstructural scale.

3.2. Water Absorption of the Untreated and Treated Soils

The water absorption of the untreated and treated gypseous soils was investigated, and the results show that the rate of water ingress was influenced by the gypsum content of the gypseous soils. For the untreated soils, the water absorption was observed to reduce initially when subjected to cyclic wetting and drying before increasing at the end of the 6th cycle, as shown in Figure 4. The initial reduction in water absorption is due to the restructuring of the soil matrix because of the removal of water from the pores, as also observed by [48–52]. At the initial wet–dry cycle, the untreated gypseous soils showed lower water absorption due to reduced void spaces induced by shrinkage. However, as the wetting and drying continued, the dissolution of the gypsum particles within the soil matrix increased the porosity of the untreated gypseous soils over that of the untreated non-gypseous (soil 1). Culminating in higher water content of 32% for soil 2, 36% and 35% for soil 3 and soil 4, respectively, at the end of the 6th wet–dry cycle, as shown in Figure 4. For soil 1, the development of the macro cracks was observed at the 5th cycle, and this was slower when compared to that of the untreated gypseous soil (soil 2 with 5% gypsum) which had desiccation cracks occurring at the 4th cycle. For the untreated soil 3 and 4, the desiccation cracks occurred at the 3rd and 4th cycles for the gypseous soils with 15% and 25% gypsum content, respectively, as shown in Figure 4a.

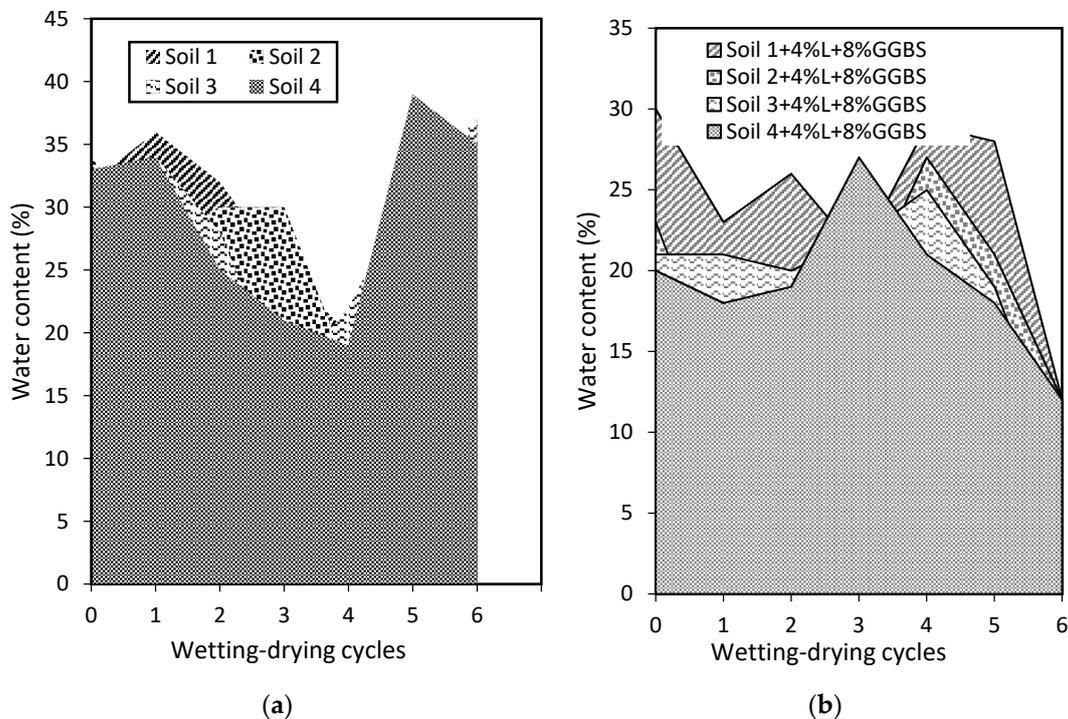


Figure 4. Water content trend during wetting–drying cycles: (a) untreated; (b) 4% lime and 8% GGBS treated.

On the other hand, the water absorption of the treated gypseous soils plotted against the wet–dry cycles is shown in Figure 4b. The treatment of the gypseous soils with 4% lime and 8% GGBS reduced the water absorption of the treated gypseous soils. This improved water absorption can be attributed to cementation and bonding of soil particle, which resulted in reduced porosity and increased resistance to tension cracks under wet–dry

cycle conditions. It is important to note that the reduction in water absorption followed increasing gypsum content from 23%, 21%, 20%, and 12% in the order of reducing gypsum content and can be linked to higher cementation. Higher gypsum content in the presence of 4% lime and 8% GGBS with contribution of CaO from gypsum makes more CSH available and increased bonding, which improves resistance to tensile stresses and crack development under wet–dry cycles. This improvement in water absorption agrees with the study by [53].

3.3. Effects of Gypsum Content and pH

The pH of treated soils is an important parameter influencing the rate of pozzolanic activities in the soil because sufficient alkalinity enhances the development of strength yielding compounds and enables higher cation exchange with early strength gain. The untreated control sample (Soil 1) shows slightly high alkalinity with a pH of 8.2 due to the bentonite content. The results of the pH test of the gypseous soils plotted in Figure 5 show that the pH decreased with increasing gypsum content due to the neutrality of gypsum. Before subjecting the untreated gypseous soils to wet–dry cycles, the pH of soil 1 was 8.18, while that of soil 2 was 7.2. Soil 3 and soil 4 had an initial pH of 6.89 and 6.86, respectively. Upon subjecting the samples to wet–dry cycles, the pH of the gypseous soils was observed to reduce with increasing gypsum content and wet–dry cycles, as shown in Figure 5. The reduction in alkalinity can be linked to the dissolution of the gypsum within the matrix of the gypseous soils following repeated wetting and drying. However, for the treated soils, treating with 4% lime and 8% GGBS increased the pH of the soils due to the high alkalinity of the lime. Additionally, the increased alkalinity was observed to be approximately constant (7–8) during wet–dry cycles. There was no significant reduction in the pH, as shown in Figure 6a–f. The improved alkalinity by the addition of 4% lime and 8% GGBS contributes simultaneously to increased SiO₂ and CaO supply and supports continuous development of cementitious products, which contributed to improved interparticle bonds and subsequent resistance to desiccation cracks as reported in [34].

3.4. Microstructural Characteristics

The morphological transformation of the treated soils using the combination of gypsum, lime and GGBS on the kaolin–bentonite mixtures can be seen in the micrograph presented in Figure 7a–d. The SEM results of the untreated non-gypseous soil confirms a more porous mass when compared with the untreated gypseous sample. The soil particles of the non-gypseous soil can be seen loosely bonded by cohesion of the grains rather than being cemented. This less dense matrix is a major contribution to the lower UCS even before the samples were subjected to wet–dry cycles and the subsequent lower resistance against desiccation cracks with increasing wet–dry cycles of the untreated non-gypseous soil. This state of the soil is largely a function of the lack of binding compound (CSH and CAH). Comparing the untreated non-gypseous soil (soil 1) with the untreated gypseous soils, it is clearly seen that a denser matrix ensued from the addition of gypsum due to slight development of cementation from the addition of gypsum and the Si–Al content in the soil following slightly higher calcium oxide availability with increasing gypsum content, which reduced the porosity of the gypseous soils. Furthermore, following treatment with lime and GGBS, a denser matrix is observed with the addition of 4% lime and 8% GGBS to the gypseous soil, as shown in Figure 7c. The higher pH of the soils and higher calcium and silicate availability from lime addition improved the pozzolanic activity as reported by [49,54], which resulted in less porous mass since the particles were more glued together by cementation and by the effects of cation exchange. A higher calcium content due to the combined effects of gypsum, lime, and GGBS shows increased binding capability and improved strength. The more flake-like CSH and CAH gels were formed as reported by [55–58], resulting in a denser matrix with far less pores compared to treated non-gypseous sample. The SEM result of the treated gypseous soil in Figure 7d shows no

visible ettringite formation which can be attributed to the rather early age of the treated gypseous soil and hence, significant reduction in swell potential, as shown in Figure 8a,b.

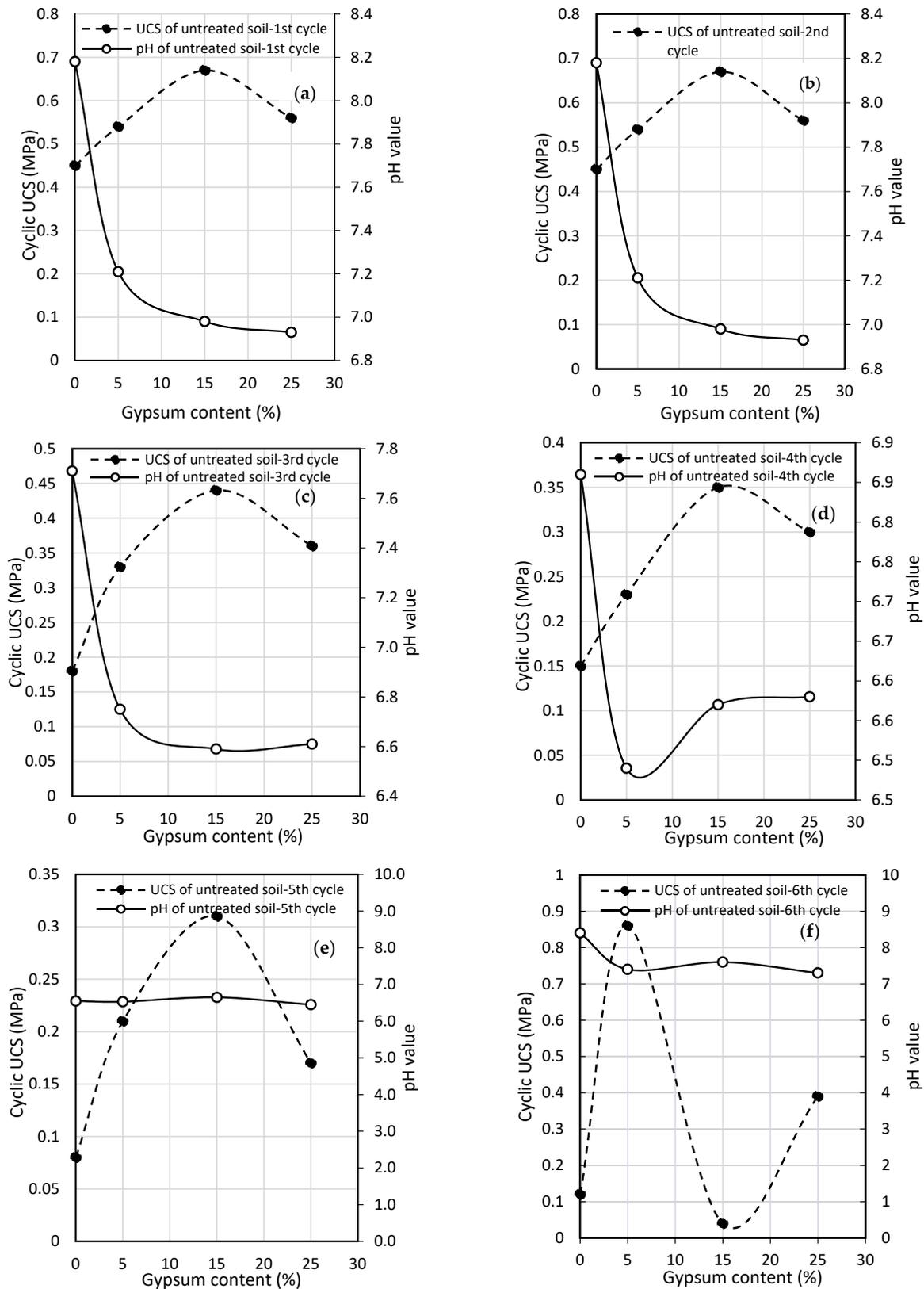


Figure 5. Cyclic UCS and pH of untreated soil–gypsum mixtures: (a) 1st cycle; (b) 2nd cycle; (c) 3rd cycle; (d) 4th cycle; (e) 5th cycle; (f) 6th cycle.

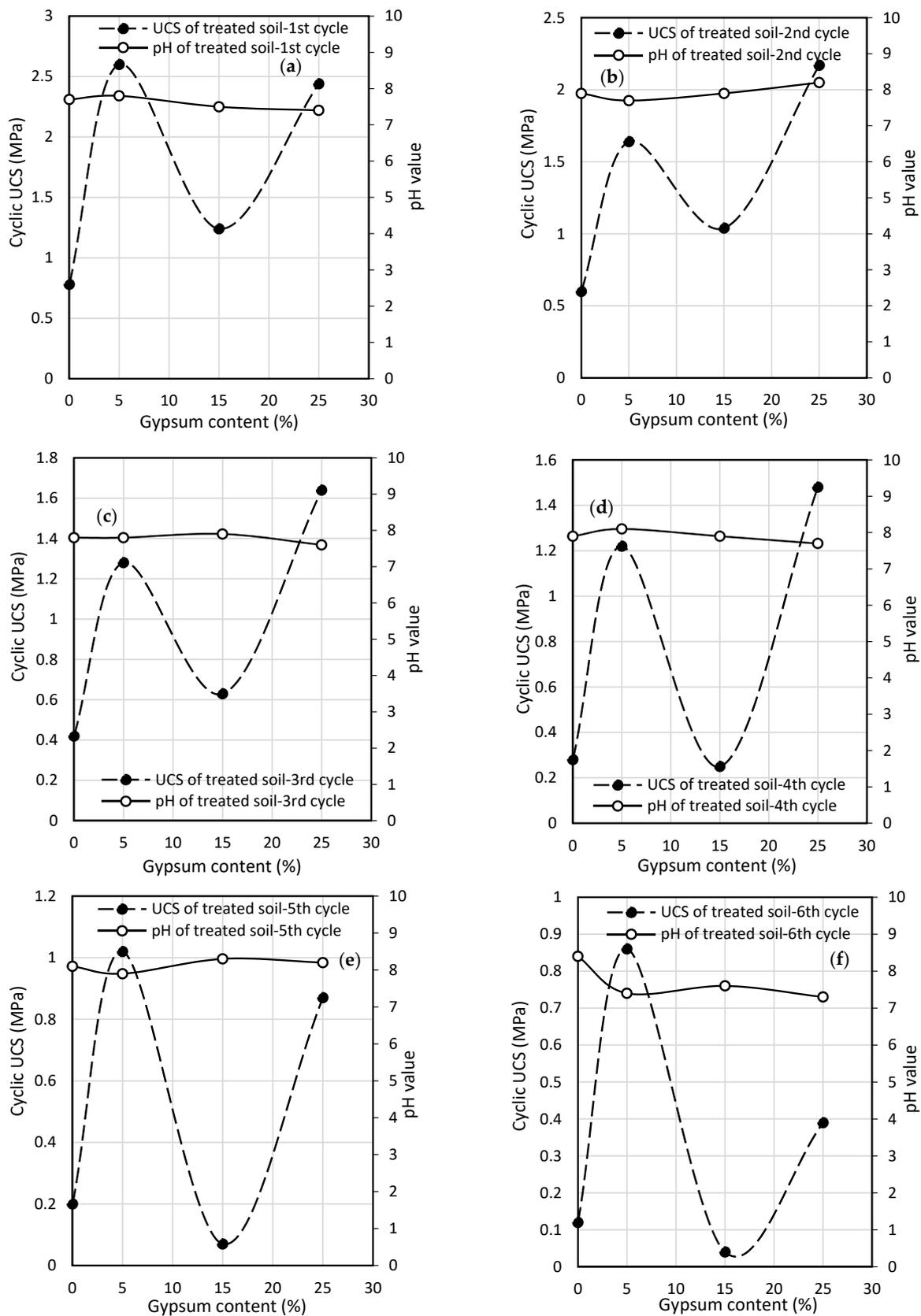


Figure 6. Cyclic UCS and pH of treated soil–gypsum mixtures: (a) 1st cycle; (b) 2nd cycle; (c) 3rd cycle; (d) 4th cycle; (e) 5th cycle; (f) 6th cycle.

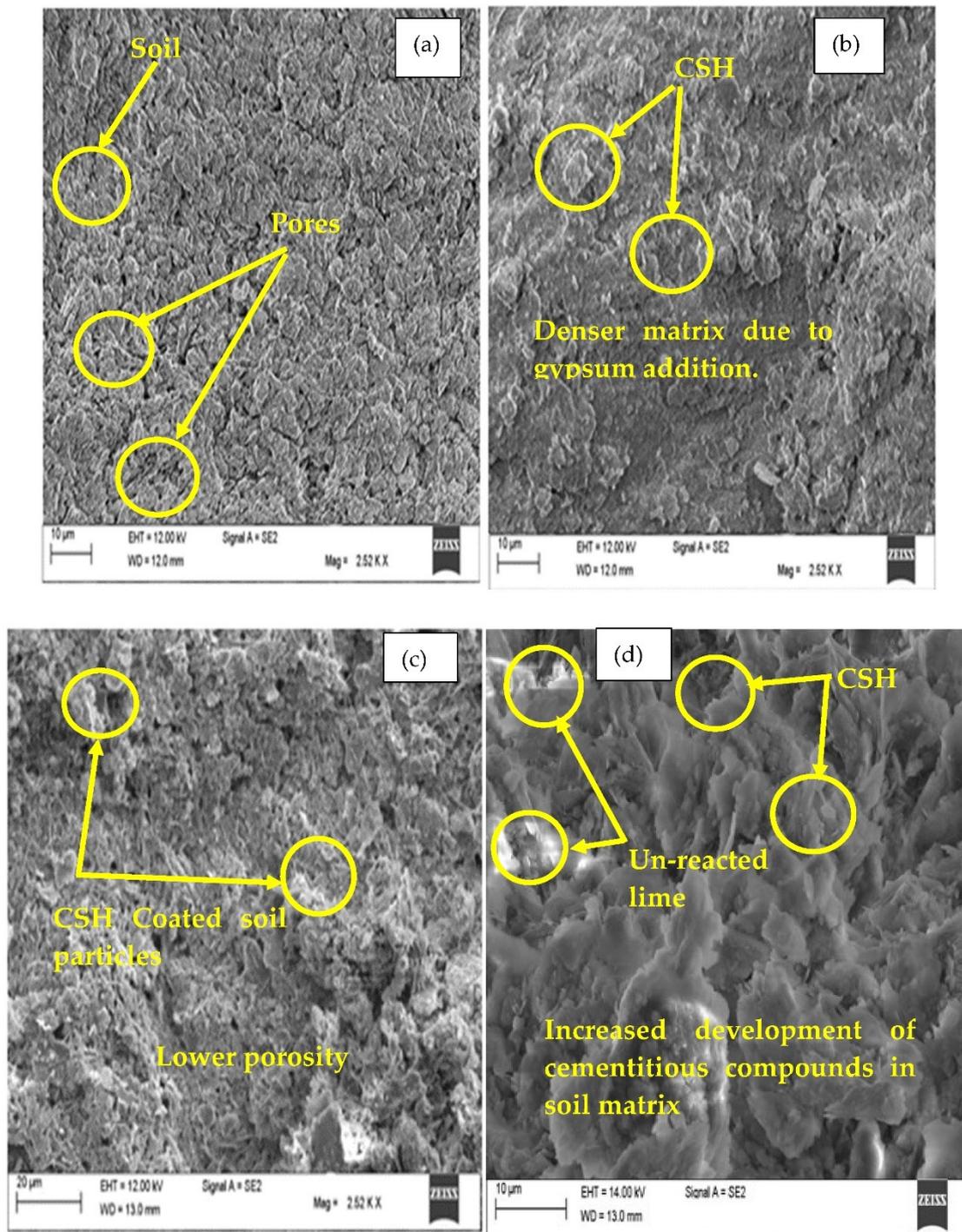


Figure 7. Untreated and treated gypseous soils: (a) untreated non-sulphate soil; (b) lime-GGBS-stabilised non-sulphate soil; (c) lime-GGBS-stabilised sulphate soil; (d) low-sulphate-bearing soil stabilised by lime-GGBS combination.

In addition, the results plotted in Figure 8a,b show that the inclusion of gypsum to the natural soil (soil 1) decreases the swell potential from 69% to 23% for the untreated gypseous soils after the soaking period due to the development of cementitious bonds at 7 days. Further reduction in swell potential was recorded upon treatment with 4% lime and 8% GGBS due to the enhanced cementation effect in the presence of lime and GGBS. The swell potential further decreased from 69% to 1.25% for soil 1, and from 30% to 1.7% for soil 2, and from 23% to 3.4%, and 25% to 2.4% for soil 3 and 4, respectively.

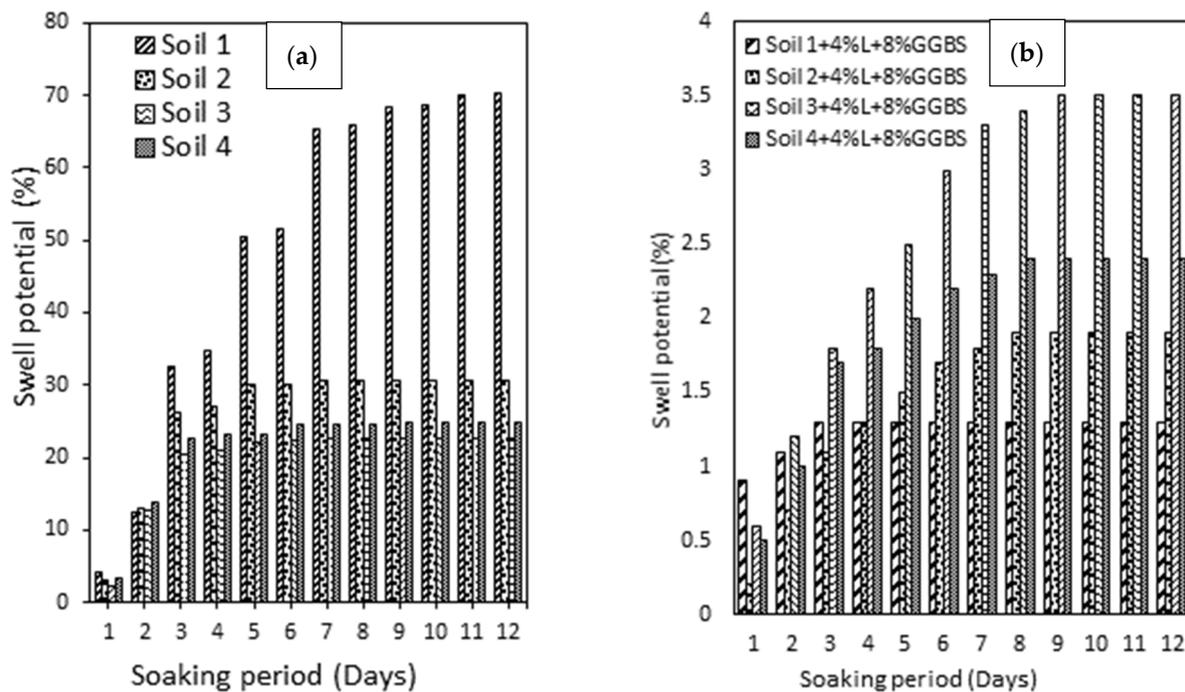


Figure 8. Swell potential of treated and untreated gypseous soils: (a) untreated gypseous soil; (b) lime-GGBS-treated soils.

4. Conclusions

In this study, the addition of lime and GGBS mixtures to kaolin–bentonite soil under cyclic wet–dry conditions has been investigated. This study simulates early age behaviour of stabilised sulphate-bearing pavement subgrade materials. Subgrade materials are commonly evaluated using the UCS of the stabilised soils after 7 days. Thus, the performance of the treated soils during the early stages of strength development becomes a parameter for measuring the acceptability of the stabilisation programme. Additionally, in arid regions where these gypseous soils are very common, and in tropical regions following higher temperatures from changing climatic conditions, it is therefore of practical importance to investigate other pertinent qualities of stabilised gypseous subgrade materials such as their capability to resist crack development and expansion due to rapidly fluctuating field conditions such as high temperatures and flooding at this early age. The use of lime and GGBS as a binder in improving the strength of gypseous clays and the durability of the treated clays when subjected to wet–dry cycles has been evaluated. From the results of the experiments, the addition of gypsum to lime causes an initial increase in the UCS and a reduction in swell of the soil with increasing gypsum content. However, when the untreated gypseous soils were subjected to wet–dry cycles, the UCS was observed to reduce with increasing gypsum content due to the dissolution of gypsum particles with the pores of the gypseous soils by water ingress. In addition, the resistance to desiccation cracks was lower for the untreated gypseous soils with higher gypsum content, which increased water absorption at the end of the wet–dry cycles. However, the effectiveness of the lime-GGBS treatment was observed in the treated soils from enhanced cementation, which resulted in increased UCS as expected. The application of 4% lime and 8% GGBS improved the pH of the treated soils during the wet–dry cycle and enhanced the resistance to desiccation cracks and water absorption. Additionally, the lime-GGBS-treated soils showed lower swell potential at the end of the wet–dry cycles. From the results of this study, the following conclusions can be made.

- The use of 4% lime and 8% GGBS is an effective binder combination for improvement of gypseous soils with 5–25% gypsum content.
- The addition of lime and GGBS to gypseous soils improves the early age characteristics of the treated soil under cyclic wet–dry conditions and is a suitable treatment for

gypseous materials under the influence of environmental changes faced by subgrade materials in tropical regions.

- Lime and GGBS treatment is effective in improving the water absorption of treated gypseous soils under wet–dry cycles. The application of GGBS and lime in stabilisation of a sulphate-bearing subgrade soil proved effective in improving strength and reducing swell of treated sulphate soils.
- The addition of lime and GGBS improves the microstructural properties of treated gypseous soils through the precipitation of cementitious compounds, which increases the strength of interparticle bonds, reduces porosity, and improves resistance to development of desiccation cracks under wet–dry conditions.
- It is also logical to conclude from this study that extending the wet–dry cycle period may have eventually led to a lower UCS value for soil 4 with 25% gypsum following further dissolution of gypsum as envisaged.

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Abbreviations

UCS	Unconfined compressive strength
pH	Potential of hydrogen
GGBS	Ground granulated blast furnace slag
PFA	Pulverised fuel ash
SEM	Scanning electron microscopy
CSH	Calcium silicate hydrate
CAH	Calcium aluminate hydrate
OLC	Optimum lime content

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