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The role of moisture content, mixing method and sample size on the swelling of sulfate soil stabilised with lime-silica fume blend.

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Abstract: Chemical soil stabilisation, a conventional soil treatment technique, is a function of several variables including the mineralogical compositions of soil, the oxide contents of the stabiliser, the sulfate concentration of soil, and the water content used for compaction, among other variables. This paper reports an experimental study investigating the impact of variation in the moisture compaction content, mixing method and specimen size on sulfate soil stabilisation with the co-addition of lime (L) and silica fume (S). A series of artificially gypsum-dosed kaolin specimens were prepared using a binder composition of 3L-7S, two different moisture contents (31 and 33%), two different mixing methods (dry mixing method-DM and slurry mixing method-SM) and two different specimen dimensions; one with 100 mm in height and one with 19 mm in height. Thereafter, a set of physicomechanical engineering tests including the unconfined compressive strength (UCS) test, linear expansion test and swelling potential test were conducted to examine their physical and mechanical behaviour. The finding of this study indicated that the use of SM instead of DM induced a compromise on both the expansion and UCS performance due to the clumping and the heterogeneity of the formed hydrates. As for the moisture content variation, the result showed that the higher moisture content of 33% yielded a better expansion and lower UCS performance due to the enlargement of voids which reduces the robustness against loading and facilitates the accommodation of ettringite.

Keywords: (Ettringite, soil stabilisation, calcium-based stabiliser, compressive strength, expansion)

Introduction

Soils specifically those comprised mainly of clay minerals, in their natural state, are typically characterised as weak and low-grade construction materials with a reversible shrink-swell behaviour [1], as their properties are influenced by the variation of moisture content [2]. Hence, the construction of engineering structures on such soil is a challenging task and can be harmful [3], facilitating crack formation, differential settlement, and sudden failure due to their changing behaviour [4], thus inducing economic concerns. For example, the annual damage cost associated with the reversible shrink-swell behaviour of expansive soil exceeds £400 million in the UK [5], and 15 billion in the USA [6]. However, replacement of the sub-grade layer with high-bearing capacity soil is considered costly and time-consuming [7]. For this reason, engineers tend to stabilise and improve the physicomechanical properties of the local soil materials by using cementitious materials including calciumbased binders such as lime (quicklime or hydrated lime) and Portland cement, of which lime is the most preferred binder [8], due to its effectiveness in the stabilisation of soil, and its lesser price related to Portland cement.

The use of lime in soil improves the physico-mechanical properties through two key reaction mechanisms: short-term reactions (including cation exchange and flocculation and agglomeration of soil particles), and long-term reactions (pozzolanic reactions) [9]. Upon the addition of hydrated lime to the soil in the presence of moisture, the hydrated lime first dissolves into calcium ions (Ca^{2}) and hydroxide ions (OH). The Ca^{2+} ions fix to the outer surface of soil particles, substituting the exchangeable cations through replaceability order of $\text{Na}^+ \leq \text{K}^+ \leq \text{Ca}^{++}$, in which higher valence cations substitute those lower valence cations [10]. This cations substitution balances the electronic charge of soil particles, induces flocculation of soil particles [11], and promotes different particle arrangements [12]. Besides, the hydroxide ions (OH) cause a significant increase in the alkalinity value up to (pH=12.4), thus creating a corrosive alkaline environment, which in turn aids the aluminate and silicate sheets of soil particles to release silica and alumina ions. This, thereafter, initiates the pozzolanic interactions between the Ca^{2+} from the binder, silica and alumina from soil, and water, thereby, forming hydrates such as calcium silicate hydrate (C-S-H), and calcium aluminate hydrate (C-A-H). These hydrates interlock and improve the physico-mechanical properties of the soil-lime mixture. However, in the presence of sulfate, an alteration in the reaction mechanism occurs due to the formation of ettringite from the reaction between calcium, alumina, sulfate, and water [13]. This mineral is an issue in sulfate soil stabilisation, as it induces a massive expansion [14]. Apart from that, lime production requires enormous energy (4000 MJ per tonne) [15], with corresponding carbon dioxide emissions of 800 kilograms per tonne released to the atmosphere [14]. Therefore, the use of industrial by-products (pozzolans) such as silica fume, has been recently encouraged due to its pozzolanic reactivity and its superior efficiency in the restriction of the swelling of gypsum-bearing soils [14, 16-26].

In the literature, Ghorbani et al., 2015 [17] observed a significant swelling reduction by using a binary blend of lime-silica fume (L-S) as a stabiliser for gypsum-based sandy soil with a gypsum content of 25%. Ebailila et al., [14] examine the viability of L-S blends at different binder contents on the suppression of artificial gypseous kaolin and concluded that a 3L-7S is the optimal bended combination for restricting the nucleation of ettringite in kaolin soil made with the gypsum concentration of 9%. Recently, Ebailila et al., 2022 [26] compares the efficiency of 3L-7S with that of a binary binder containing 3% lime and 7% ground granulated blast-furnace slag (3L-7GGBS) on the expansion of gypseous kaolin. This study concluded that both blended binders (3L-7GGBS and 3L-7S) are effective in sulfate soil stabilisation, of which 3L-7GGBS was the optimum for the UCS while the 3L-7S was the best for the expansion.

Given the description above, it can be stated that the use of L-S in sulfate soil stabilisation is beneficial. However, studies concerning the effect of moisture content, mixing method and sample size variation on sulfate soil treated with the co-addition of Lime and silica fume, are still insufficient. Therefore, complementary to the existing knowledge, this study aimed at examining the co-effect of L-S on gypseous kaolin specimens made with different mixing methods, different moisture contents and different specimen sizes, to propose the optimum mixing and optimum moisture compaction condition. To do so, a set of samples mixed using different moisture contents (31% and 33%) and different mixing methods and produced with different dimensions, were evaluated in terms of the UCS, linear expansion and swelling potential.

Methodology

1- Materials

The raw materials used in this research included one target soil material (artificial gypseous kaolin soil-K9G made from Kaolin-K and gypsum-G), two cementitious materials (lime-L and silica fume-S) and tap water. **Table 1** and **Table 2** show the oxide and physical properties of the raw materials, respectively, whereas **Fig. 1** and **Fig. 2** present the x-ray diffraction of kaolin and the sieve analysis results of the raw materials, respectively.

Oxides	K	L	S
CaO	< 0.01	71.56	0.2
MgO	0.21	0.58	0.1
SiO.	47.32	0.67	98.4
ALO.	35.96	0.07	0.2
Na ₂ O	0.07	<0.02	
P_2O_5	0.12	0.03	0.03
Fe,O,	0.69	0.05	0.01
Mn2O3	0.02	0.02	
K,O	1.8	< 0.01	0.2
TiO.	0.02	< 0.01	
V-O-	< 0.01	0.02	
BaO	0.07	< 0.01	
SO.	0.01	0.19	0.1
LOI	0.1	27.4	0.5

Table 1: Oxide characteristics of the raw materials.

Table 2: Physical properties of the raw materials used in this study.

Properties	K		S
Bulk density $\left[\frac{\text{kg}}{\text{m}^3}\right]$		480	300
Particle density (Mg/m^3)	2.1	2.82	3.15
pH Value	5.37	12.62	
Colour	White	White	White

The K9G was prepared by mixing industrial kaolin soil (K) with 9%, by the dry weight, of gypsum (G) in a mixer for about 3 minutes, as commonly adopted for sulfate soil stabilisation-based research [13,14]. The K used was in the texture form of white powder and was sourced from Potterycrafts Ltd., Stokeon-Trent, UK. The XRD analysis revealed that K is composed of kaolinite minerals and quartz, while the sieve analysis indicated that K composes of 12% clay, 60% silt and 28% sand. The characterisation tests indicated that the K has a liquid limit (LL), plastic limit (PL) and plasticity index (PI) of 56.7%, 33.3% and 23.4%, respectively, so the K is classified as a medium-graded sandy SILT.

Fig.1: The XRD pattern of kaolin soil

Fig.2: Sieve analysis curve for the raw materials.

The G used was a calcium sulfate dihydrate with a white powder form; it was acquired from Fisher Scientific Ltd, Loughborough, Leicestershire, UK. The L used was quicklime with an off-white powder form and calcium oxide percentage of 71%; it was sourced from Tarmac Cement and Lime Company, Buxton Lime and Powders, Derby, UK. As for the S, it was a commercially reactive micro-silica with a light grey powder form and silicon dioxide concentration of 98.4%; it was sourced from Tarmac Cement and Lime Company, Buxton Lime and Powders, Derbyshire, Derby, UK.

2- Mix design

To evaluate the independent role of the variation in the moisture compaction content, mixing method and specimen dimensions on sulfate soil stabilisation with the co-addition of lime and silica fume binder, the methodology of this research study involved using; 1) one target soil material (artificial gypseous kaolin-K9G); 2) a constant blended binder content (10% by the weight of the target soil material); 3) a constant blended binder composition (lime-silica fume) of 3L-7S; 4) two different moisture compactions contents; 5) two different mixing methods; and 6) two different specimens.

The blended binder composition was made of 30% lime and 70% silica fume, in line with the previous study [14]. The moisture compaction contents adopted were 31% and 33%, corresponding to 1.1 and 1.2, respectively, of the corresponding standard proctor optimum compaction condition. This was selected; 1) to ensure the soil material is compacted wet of OMC, as it is the typical condition for soil compaction in practice to actualise the best durability behaviour [13,14], and 2) to determine the optimum moisture compaction condition for the sulfate soil. The two mixing methods selected were mainly the dry mixing method (denoted as DM) and the slurry mixing method (denoted as SM), which differ in terms of the order of the mixing procedure. For the case of dry mixing method (DM), all the dry raw materials were firstly mixed in a dry form for about 3 minutes before the predetermined moisture content was steadily introduced and the mixing was restarted for additional three minutes, preparing for the compaction process. However, in the case of slurry mixing method (SM), firstly, the binder and water were mixed for three minutes, before the artificial gypseous soil was poured and the mixing was continued for further three minutes, preparing for the compaction process. Therefore, in total, the detailed mix proportions of the sulfate-bearing kaolin soils stabilised with the co-addition of lime and silica fume at a constant binder content examined in the experimentations can be summarised in **Table 3**.

As for the two different specimens, they were fabricated in a cylindrical form with different dimensions. The first one (referred to as a jack-compacted specimen) was cast using a prefabricated mould with internal diameter of 50 mm and internal height of 100 mm and compacted using a manual jack. This kind of specimen was used to evaluate the linear expansion behaviour using Perspex cells in line with [13,14] and the UCS performance at the end of 7, 28 and 90 days of moist curing. The second specimen (referred to as a ring-shaped specimen) was compacted using similar procedure to that of proctor compaction test and extruded manually in a miniaturized cylinder with a diameter of 76.2 mm and height of 19 mm, corresponding to the initial dimensions of the ring of the standard onedimensional odometer apparatus. This was used to evaluate the swelling potential of the control mix (K9G–3L7S–1.1MC–DM) using a standard one-dimensional odometer.

Table 3: Mix compositions of artificially gypseous kaolin specimens treated with lime-silica fume blend at a constant stabiliser content of 10% by the total weight of the soil materials.

3- Specimens preparation

3.1- Jack-compacted specimen

A total of 11 jack-compacted cylinders with an outer diameter of 50 mm and height of 100 mm were fabricated for each of the mix compositions outlined in **Table 3**. Two of these specimens were employed for measuring the linear expansion, while the nine specimens were utilised for evaluating the UCS after 7, 28 and 90 days of moist curing. For each specimen, after mixing the raw ingredients as per the selected mix design outlined in **Table 3**, the semi-paste mixture was placed into the mould and compacted by means of a jack wherein a compression force was applied in aid of a steel frame as photographed elsewhere [14]. Hereafter, the samples were kept in the mould for about three minutes, permitting the stability of samples. Consequently, the specimens were extruded using a plunger, and wrapped individually in several runs of cling film to reduce moisture evaporation and carbonation, as well as regulate humidity, in line with [13,14]. Eventually, the compacted samples were further kept in a sealed plastic container and stored in the laboratory at $20\overline{+}2^{\circ}C$, allowing for moist curing until the date of testing.

3.2- One-dimensional odometer specimen

Two ring-shaped specimens were prepared in this study for the control mix to evaluate the swelling potential and form a relationship between the linear expansion and the standardised swelling methods (ASTM D4546). For each specimen, all the dry raw materials, as per the mix composition outlined in **Table 3**, were firstly mixed in a dry form for three minutes, before the moisture was included and the mixing was continued for extra three minutes. Thereafter, the semi-paste was poured into the proctor compaction mould (with height of 116 mm and diameter of 102 mm) in three layers, of which each layer was compressed by the application of 25 using a 2.5 kg rammer. Subsequently, the ring-shaped sample was extruded by pushing the ring of the oedometer device into the compacted mixture, wrapped and stored under a similar condition to that of jackcompacted specimens for 7 days of moist curing.

4- Testing method

4.1- UCS

The UCS test was conducted on three specimens per mix composition at the end of 7, 28 and 90 days of moist curing. The test was operated in accordance with [27], using a Hounsfield compression machine equipped with a self-levelling device to guarantee a uniaxial load application. The load application was performed at a continuous compressed strain ratio of two mm per minute, and the average value of the three specimens was adopted as the presentative UCS.

4.2- Linear expansion

The linear expansion was conducted, in line with DS EN 13286-49: 2004 [28], on two jack-compacted specimens per mix over a 200-days water soaking period, using Perspex cells, as commonly adopted for soil stabilisation-based research [8,14,26,29-31]. Immediately after seven days of curing, the 10 mm-top and 10 mm-bottom part of the samples were unwrapped and accommodated individually into the Perspex cell as presented in **Fig. 3**. Afterwards, the dial gauge was adjusted to zero and the water was introduced through the upper inlet until the 10 mm-bottom of the samples was immersed in water, allowing for swelling as shown in **Fig.4**. The gauge reading was then recorded daily for 28 days, and then random reading was recorded for the remaining period. Ultimately, the ratio of the dial gauge reading (mm) to the original height (100 mm) of the specimens was calculated, and the mean of the two swelled samples was adopted as the representative linear expansion.

Fig.3: The Perspex cell set-up used for expansion (swelling) measurement.

Fig.4: Linear expansion specimens under expansion measurement.

4.3- Swelling potential

The swelling potential test was performed on two ring-shaped specimens for the control mix after 7 days of moist curing. The test was operated using the standard one-dimensional odometer equipment, according to ASTM D4546. After calibration with an initial pressure of 2.75 kPa (cap load) and adjusting the dial gauge to zero, the water was introduced until the specimens were completely submerged in water as schematically shown in **Fig. 5**. Thereafter, the specimens were allowed to swell for a prolonged soaking period of 100 days, and the dial gauge reading was monitored regularly till no vertical displacement was recorded. Finally, the ratio of the dial gauge reading (mm) to the original height (19 mm) of the specimen was calculated for both specimens, and the mean value in percentage was used as the presentative swelling potential (%).

Fig.5: The schematic diagram of swelling potential specimen in the standard one-dimensional odometer apparatus.

Results and discussion

1- UCS

Fig.6 shows the influence of change in moisture content and mixing method on the UCS evolution of the artificially gypseous kaolin (K9G) samples treated with lime-silica fume blend of 3L-7S. In this perspective, the UCS revealed a progressively increasing trend as the curing age increases, confirming the classical UCS development trend of soil stabilisation. This development trend is normally assigned to the cation exchange and flocculation-agglomeration of soil particles (short-term interaction) and the pozzolanic reactions (long-term interaction) between the soil and binder, which eventually form hydrates such as calcium silicate hydrate (C-S-H) and calcium aluminate hydrate (C-A-H). These hydrates, whose kinetics is dependent on the mineralogy of the soil and the available quantities of the concerned oxides within the binder, crystalline with time, fill the pore space [10] and interlock the soil system. This consequently forms a stiff soil matrix and enhances the mechanical performance such as the shear resistance and the UCS performance [32].

On increasing the moisture compaction content from 31% (K9G-3L7S-1.1MC-DM) to 33% (K9G-3L7S-1.2MC-DM), the stabilised specimens exhibited a compromise on the UCS. This compromise on UCS was further increased corresponding to the change of mixing method from DM (K9G-3L7S-1.1MC-DM) to SM (K9G-3L7S-1.1MC-SM). This, therefore, implies that the increase in moisture content and the use of the SM are not favourable for a higher degree of soil stabilisation. The reduction in UCS induced by the moisture content increase is probably owing to that the increase in moisture induces a lower inter-particle friction and a

relatively poorer interlocking, both of which induce larger inter-particle void spaces [33]. Thereby, this void enlargement negatively affects the interlocking of the soil matrix by increasing the porosity, thus, minimising the robustness against loading [34,35]. In addition, the nucleation and growth of a higher quantity of ettringite, is also a contributing factor for the reduction in UCS [36,37], as the nucleation of a higher ettringite quantity induces resistance in compaction and facilitates the formation of extensive cracks within the system, both of which induce an increase in porosity and thereby induce a compromise on the UCS. As for the reduction in the UCS corresponding to the change of mixing method from DM to SM, it is supposed to be caused by the clumping of the cementitious ingredients (lime and silica fume) during the mixing and the poorer distribution of the hydrates, both of which induce poorer interlocking, and lowered UCS.

2- Volume change behaviour.

2.1-Effect of variation of water content and mixing method.

The effect of water compaction content and mixing procedure on the expansion of gypseous kaolin treated with a lime-silica fume blend of 3L-7S, is presented in **Fig. 7**.

Fig.7: The linear expansion plots of K9G-3L7S specimen mixed at different mixing methods and moisture contents.

Accordingly, the utilisation of higher water compaction content (1.2MC as against 1.1MC) shows a beneficial impact on the expansion behaviour, where the total expansion of K9G-3L7S was reduced from 4% to 2.3%, as the moisture content increased from 31% (1.1MC) to 33% (1.2MC), respectively. Conversely, the utilisation of SM (slurry mixing) instead of DM (dry mixing) negatively affects the expansion trend, where the total expansion magnitude was increased

from 4% for the case of DM to about 5% for the SM, representing about 23% increase in the expansion. Therefore, it can be stated that using higher water compaction content is beneficial in terms of the expansion, while the use of the SM is not recommended. The possible rationale for the reduction in expansion induced by the higher moisture content could be the increase in the inter-particle void pores [33]. At higher water level (33%), the stabilised samples would not be at the maximum dry density, thus a higher volume of voids would be available for the nucleation and the growth of ettringite, and thereby a lesser swelling magnitude would be induced [38]. As for the increase in the expansion induced by the slurry mixing method, it is expected to be due to the poorer interlocking as a result of the clumping of the cementitious ingredients during the mixing process and the poorer distribution of pozzolanic hydrates within the stabilised system.

2.2- Effect of specimen size and testing approach.

The swelling potential trend measured using the standard one-dimensional odometer test for the gypsumdosed kaolin specimen stabilised with 3L7S is plotted in **Fig.8**, in comparison with its expansion counterpart measured using Perspex cell.

Fig.8: The linear expansion and swelling potential plots of K9G-3L7S mixed using the dry mixing method and compacted at 1.1MC.

As expected, the mix composition of K9G-3L7S-1.1MC-DM exhibited a higher swelling potential of 4.7%, relative to that of 3.9% expansion obtained using Perspex cell. This increase in swelling potential can be attributed partly to the densification degree of the specimen, and partly to the specimen confinement matter during the test. As discussed earlier, the one-dimensional odometer specimen was compressed on the matter of proctor test in which 25 blows were applied using a 2.5 kg rammer, as against that of the linear expansion specimens which were compacted using a simple hydraulic jack. This implies that the samples used for the swelling potential are likely to be compressed at a higher dry density, thus the volume of voids is lower than those of the linear expansion specimen, thereby lesser space is available for accommodating the ettringite and in turn a higher swelling potential would be induced. As for specimen confinement condition, the perimeter of the odometer specimens was confined by the ring, thus the displacement (swelling) of the specimens occurred only in the vertical direction, thereby a higher swelling potential was recorded. On the contrary, the perimeter of the linear expansion specimens was unconfined, thus the expansion occurs in both the vertical direction and perimeter direction. Eventually, it can be inferred that the volume change of gypseous kaolin treated with lime and silica fume is also a function of compaction degree and sample confinement matter during the experimentation analysis.

Conclusions

The role of moisture compaction content, mixing method and specimen size on the UCS performance and swelling behaviour of sulfate soil stabilisation with the co-addition of lime (L) and silica fume (S) has been examined in this research study. Accordingly, the following specific conclusions can be outlined:

The utilisation of a moisture compaction content of 1.2 of the corresponding standard Proctor optimum compaction condition yielded a compromise on the UCS, relative to that of 1.1 MC, although such a relatively higher moisture content reduces the ultimate expansion magnitude. This is attributed to the lower interparticle friction and the enlargement of voids within the system, both of which reduce the robustness against loading and facilitate the accommodation of ettringite.

The slurry mixing method, in which the binder was initially mixed with water and then the soil was incorporated, yielded a compromise on both the UCS and linear expansion due to the clumping of the binding constituents and the poorer distribution of the hydrates formed from the binder hydration. Hence, slurry mixing is not suggested for a better soil stabilisation.

The volume change of gypseous soil samples treated with a binary blend of 3L-7S is a function of densification degree and testing approach, where the comparison between swelling potential measured using a onedimensional odometer test and linear expansion test indicated a higher swelling potential magnitude due to the higher degree of specimen densification and the confinement of specimen's perimeter under the testing.

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