Lime Stabilisation of Organic Clay and the Effects of Humic Acid Content

N.Z. Mohd Yunus¹, D. Wanatowski² and L.R. Stace²

¹Department of Geotechnics & Transportation, Universiti Teknologi Malaysia, Skudai, Malaysia

²Nottingham Centre for Geomechanics, Faculty of Engineering, University of Nottingham, United Kingdom

E-mail: dariusz.wanatowski@nottingham.ac.uk

ABSTRACT: The effectiveness of lime as a chemical additive for the stabilisation of organic clay is considered uncertain, especially in the long term. The presence of humic acid is believed to be the main detrimental constituent of organic matter that renders lime stabilisation inefficient. In this paper, the amount of humic acid that may render lime stabilisation inefficient was carefully investigated. Artificial organic clay, prepared by mixing commercial kaolin and various amounts of humic acid (0.5%, 1.5% and 3.0%) was treated with hydrated lime. The strength properties of lime-treated organic clays were examined by unconfined compressive strength (UCS) and drained and undrained triaxial compression tests. Curing periods of 7, 28 and 90 days were chosen as key points to monitor the evolution and the effect of the stabilisation process on lime-treated specimens. Overall, the development of physical and engineering properties of lime-treated organic clay was most affected when the humic acid content in clay exceeded 1.5%.

Keywords: Organic clay, humic acid, lime stabilisation, shear strength.

1. INTRODUCTION

In contrast with lime stabilisation of inorganic clay, reactions between lime and clay are not efficient when organic matter is present in clay minerals. There is evidence in the literature showing that the presence of high concentrations of organic matter in clay can diminish the chemical reaction between lime and clay minerals and can have detrimental effects on engineering properties of soil (e.g. Kuno et al. 1989, Hebib and Farrell 2003; Petry and Glazier 2004; Puppala et al. 2007; Chen et al. 2009).

In particular, humic acid is a well known constituent of organic matter with the potential to disrupt the soil stabilisation process. In fact, it has been reported that, more than 1% humic acid content in clay may render lime stabilisation process ineffective (Huat et al. 2005; Koslanant et al. 2006; Harris et al. 2009; Zhu et al. 2009). This is because humic acid interferes with the pozzolanic reaction process between lime and clay by hindering or retarding it. To elaborate further, it causes a reduction in the pH of the mixtures pore solution. The effect of a highly alkaline environment (if permitted) is that it prolongs the dissolution of silica and alumina. Nonetheless, the effects of organic matter, especially humic acid, on the strength development of organic clays are not fully understood yet. Further study is still required to quantity the effects of humic acid on physical and engineering properties of lime-stabilized organic clay.

This paper presents the results of experimental study carried out on artificial organic clay with different humic acid contents. The effects of humic acid on selected engineering properties of limestabilised organic clay are investigated. The results obtained from unconfined compressive strength and triaxial tests carried out under undrained and drained conditions are discussed and summarized.

2. MATERIALS

Artificial organic clay, used in this study, was prepared by mixing commercial kaolin with 0.5%, 1.5% and 3.0% commercial humic acid ranges from 0% according to dry mass of kaolin. Hydrated lime (5%, 10% and 15%) was used to treat both inorganic (i.e. 0% humic acid) and organic clays. The results of index testing carried out on inorganic and organic clays are summarized in Table 1.

The chemical elements present in each type of soil are listed in Table 2. It should also be pointed out that the humic acid was mainly composed of carbon (52-62%), oxygen (30-33%), nitrogen (3.5-5%) and hydrogen (3-5.5%). Thus, the effect of humic acid on chemical elements can be detected from the increasing amounts of carbon and oxide ions. As a result, the amounts of pozzolanic materials (silica and alumina) in the clay decrease. Such a trend is shown in Table 2 where the increase in amounts of carbon and oxide ions and decrease in silica and alumina contents is associated with increasing humic acid content.

Table 1 Physical properties of clay with different humic acid contents

Property	K	K0.5HA	K1.5HA	K3.0HA
Liquid limit (%)	65.4	64.4	63.6	61.0
Plastic limit (%)	30.4	33.0	33.8	34.5
Plasticity index (%)	35.0	31.4	29.5	26.5
Specific gravity	2.61	2.53	2.51	2.47
pН	5.52	5.34	5.16	5.07
OMC (%)	30.6	30.9	30.6	33.4
MDD (kg/m ³)	1440	1429	1425	1404

Note: K=kaolin, HA=humic acid

	Table 2	Chemical	elements	in	tested	soils
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		Wt %		
Element	K	K0.5HA	K1.5HA	K3.0HA
С	2.12	2.8	2.74	20.16
0	47.91	50.45	54.54	48.97
Na	0.2	0.36	0.41	0.81
Mg	0.64	0.65	0.64	0.56
Al	19.43	16.94	18.47	12.8
Si	26.71	24.74	21.52	14.07
Р	0.09	0.18	0.28	0.16
S	0.02	0.05	0.12	0.42
Cl	0	0	0.01	0.19
K	2.65	3.42	0.79	0.59
Ca	0.02	0.02	0.04	0.33
Ti	0	0	0.02	0.18
V	0	0	0.01	0.01
Fe	0.21	0.39	0.4	0.74

Note: K=kaolin, HA=humic acid

3. SPECIMEN PREPARATION

All the specimens tested in this study were prepared using standard procedures described in the ASTM standard D5102-09 (2004). Initially, the specimens were oven-dried at 60°C until the constant weight was obtained. Clay with different amount of humic acid was prepared by mixing relevant amounts of dry kaolin with 0.5%, 1.5%, and 3% of humic acid by dry mass of kaolin. Mixing of dry materials was continued until a uniform appearance of the kaolinhumic acid mixture was obtained. Distilled water was then added and further mixing was performed until a homogeneous appearance of the soil paste was achieved. This paste was then used for plasticity and compaction tests.

After that, appropriate amounts of dry soil and dry lime were mixed with water according to the optimum moisture content (OMC) of the untreated clay. The process of mixing was conducted as quickly as possible to ensure that lime was not exposed to air for too long. This was necessary to avoid the carbonation process that could affect the strength characteristics of lime-treated specimens. The specimens were compacted into a mould (76 mm in height and 38 mm in diameter) at the specified moisture content to achieve the specified dry density. A small amount of grease was applied inside the brass mould to minimize friction. The specimens were then extruded from the mould and wrapped in a cling film to preserve the water content and to keep them free from carbon dioxide (CO_2). The specimens were then cured in desiccators at 20°C and with humidity more than 90% for 7, 28, and 90 days, respectively.

4. RESULTS AND DISCUSSIONS

4.1 Unconfined Compressive Strength

The results of a short term strength assessment of lime-treated clay with different humic acid contents (0%, 0.5%, 1.5%, 3%) and lime contents (5%, 8%, 10%, 15%) are shown in Figure 1. All the specimens were cured for 7 days. It can be seen from Figure 1 that the undrained shear strength (S_u) of the specimens with 5% lime content increases significantly compared to the strength of the untreated clay (i.e. 0% lime). However, the shear strength of specimens with higher than 5% lime content reduces gradually with increasing lime content, regardless of the humic acid content in the clay. Therefore, 5% was taken as the optimum lime content (OLC) for each type of organic clay. Figure 1 also shows that the shear strength of organic clay decreases with increasing humic acid content, which proves that the presence of humic acid in clay diminishes the shear strength.



Figure 1 Effect of lime content on the undrained shear strength of clay with different humic acid contents at 7 curing days

In a further assessment of the effect of humic acid on lime stabilisation, samples were tested at 0, 7, 28, and 90 curing days to ascertain the appropriate duration of stabilisation process. The abovementioned times were thought to be reasonable to assess the long term stabilisation process. Since the OLC of each specimen was identical, investigation of the development of the lime-clay reaction with time was conducted only at 5% lime content. Figure 2 illustrates the development of the undrained strength of the limetreated specimens at 0, 7, 28 and 90 days of curing. It appears that with the exception of the inorganic clay (0% humic acid) the undrained shear strength of the lime-treated clays comprising 0.5%, 1.5% and 3.0% humic acid decreased over 90 day curing period. A slight loss in strength is observed for organic clay with 0.5% humic acid, while a substantial loss in strength is evident where the humic acid content is equal to or greater than 1.5%. For example, the undrained shear strength of the specimens with 0.5%, 1.5% and 3.0% humic acid reduced from 230 kPa, 174 kPa and 140 kPa after 7 curing days to 212 kPa, 131 kPa and 96 kPa after 90 curing days, respectively. These results clearly demonstrate that lime stabilization of organic clay with a high humic acid content is not very efficient in a long term. Similar observations were reported by other researchers (Koslanant et al. 2006; Harris et al. 2009; Zhu et al. 2009). This behaviour also agrees from that observed for limestabilized inorganic clays, where the shear strength was reported to increase with curing time (Bell 1996; James et al. 2008). Furthermore, despite exhibiting a loss in the long-term strength, the undrained shear strengths of lime-treated samples at 90 curing days were still higher than those of the untreated specimens (Mohd Yunus et al. 2011). However, for practical purposes, it is not reasonable to consider lime stabilisation as successful when the long-term shear strength is uncertain.



Figure 2 Effect of curing period on the undrained shear strength of lime-treated clay with different humic acid contents

The impedance in the formation and development of cementitious products with increasing humic acid content over longer curing periods may be due to various reasons.

Firstly, the reduction in pH value at longer curing periods may be one of the reasons for reducing the shear strength values. Figure 3 shows pH values of clay with various humic acid contents treated with 5% lime at different curing periods. It can be seen that, except for the inorganic clay, the pH values of the clays were found to decrease with the increasing curing period. It should be noted that the pH of 12.40 is typically considered to be satisfactory for providing an affinity necessary for production of cementing materials. However, it can be seen that after 28 days of curing, the specimens with 1.5% and 3.0% humic acid contents had pH lower than 12.40. Consequently, the long-term shear strength could not be increased.

Secondly, bearing in mind that the dissolution of clay minerals is highly dependent on pH value, it is thought that the presence of a certain amount of humic acid made it difficult for the solution to recover from its acidified condition. The humic acid may also coat the clay particles, thereby preventing the lime from coming into contact with clay minerals during pozzolanic reactions. Therefore, dissolution of clay minerals became insufficient and production of cementitious materials is limited. Further study is currently being undertaken using Scanning Electron Microscopy to explain fully the abovementioned hypothesis.



Figure 3 Effect of curing period on pH value of lime-treated organic clay with different humic acid contents.

4.2 Triaxial Tests

The effect of humic acid on effective stress was further analysed by carrying out drained (CID) and undrained (CIU) triaxial compression tests. The results presented here, are limited to lime-treated specimens with OLC of 5% and cured for 7 and 28 days. The back pressure of 400 kPa and strain rate of 0.04%/min were applied to all the specimens under both drainage conditions. All of the specimens tested after 7 and 28 curing days were sheared at σ'_3 of 50 kPa. Selected specimens cured for 28 days were also sheared at σ'_3 of 100 kPa.

4.2.1 Undrained Behaviour

The effect of humic acid content on the undrained stress-strain behaviour of lime-treated clay at 7 curing days is shown in Fig. 4. It can be seen from Figure 4(a) and Figure 4(b), that the peak deviator stress q_p and the peak effective stress ratio $(q/p')_p$ decrease with increasing humic acid content. Furthermore, a significant decrease in the q_p and $(q/p')_p$ is observed beyond 1.5% humic acid content.

The relationships between the excess pore water pressure Δu versus axial strain, ε_a are shown in Figure 4(c). The higher humic acid content the more positive Δu was measured in the CD tests. This trend illustrates again that the shear strength of organic clay reduces with increasing humic acid content.



Figure 4 Effect of humic acid content on the undrained behaviour of lime-treated organic clay at 7 curing days: (a) $q-\epsilon_a$; (b) $(q/p')-\epsilon_a$; (c) $\Delta u-\epsilon_a$

In order to identify further the effect of humic acid content at 7 days of curing, the values of peak effective friction angle (ϕ_{p}^{r}) obtained for clays with different humic acid contents were investigated. The relationship between the ϕ_{p}^{r} and the humic acid content is plotted in Figure 5. Please note that the ϕ_{p}^{r} values were calculated based on the effective stress ratio at peak $(q/p')_{p}$ and without considering the effect of cohesion intercept (i.e. c' = 0 kPa as assumed).



Figure 5 Effect of humic acid content on the peak effective friction angle of lime-treated specimens sheared after 7 curing days under undrained conditions



Figure 6 Effect of curing time on the undrained behaviour of limetreated clay with 1.5% humic acid: (a) $q-\epsilon_a$; (b) $(q/p)'-\epsilon_a$; (c) $\Delta u-\epsilon_a$

It can be observed from Figure 5 that φ'_p decreases with increasing humic acid content. The φ'_p of lime-treated specimens with 0%, 0.5% and 1.5% were 38.1°, 37.9° and 37.1° respectively, whereas φ'_p of 30.2° was obtained for the lime-treated specimen with 3.0% humic acid content. It can be concluded that the reduction in φ'_p is more pronounced beyond 1.5% humic acid content (Figure 5).

The effectiveness of lime stabilisation in organic clays in a long term was also assessed by comparing the undrained behaviour of clay at different curing periods. The comparison of the behaviour of organic clay with 1.5% humic acid and stabilised with 5% lime is shown in Figure 6. Three curing times of 0, 7, and 28 days were chosen for the comparison. It can be seen from Figure 6(a) and (b) that the q_p and $(q/p\,{}^{\prime})_p$ of the lime-treated clay comprising 1.5%humic acid increased in the first 7 days of curing. Furthermore q_p and $(q/p')_p$ are shown to have improved in the period from 0 to $\dot{7}$ days. In contrast, a significant reductions in q_p and $(q/p)_p$ occurred at 28 curing days. Similarly, Figure 6(c) shows that the clay specimen cured for 7 days exhibited a more negative Δu during undrained shearing compared to the specimen cured for 28 days. In other words, the behaviour shown in Figures 6(a), (b) and (c) demonstrates that the strength characteristics of the organic clay reduce with increasing curing period. This observation has a significant practical implication because any decrease in the shear strength of organic clay in a long term is undesirable and it may result in a failure of a structure built on such clay.

4.2.2 Drained Behaviour

The effect of humic acid content on the drained behaviour of limetreated organic clay was also investigated. Fig. 7 shows the results obtained for the specimens with 0, 0.5, 1.5, and 3% humic acid contents after 7 days of curing. It can be observed from the q- ε_a and (q/p')- ε_a curves (Figure 7a and 7b) the higher values of the deviator stress (q_p) and the effective stress ratio $(q/p')_p$ at the peak states are associated with decreasing humic acid content. Once again, the most significant decrease in peak stress can be noted for the specimens with humic acid concentration greater than 1.5%. However, the differences between the drained responses of clay with different humic acid contents are noticeably smaller compared to those obtained under undrained conditions (see Figure 4). The changes in volumetric strain (ε_v) are illustrated in Figure 7(c). Similar behaviour was observed in each test with initial contractive response followed by dilative changes at about 6% axial strain. The higher humic acid content the more contractive behaviour was observed in the CD tests.

Similar to the CU tests discussed earlier, the effect of humic acid content at 7 days of curing was analysed in terms of the effective friction angle at the peak state (φ'_p). Figure 8 shows that φ'_p decreases with the increasing humic acid content. The φ'_p of lime-treated specimens with 0%, 0.5% and 1.5% were 31.8°, 30.8° and 30.4° respectively, while 28.6° was obtained for the lime-treated specimen with 3.0% humic acid content. These results confirm that the decrease in φ'_p was more pronounced beyond 1.5% humic acid content.

The effect of curing time on the behaviour of lime-treated organic clay (1.5% humic acid) in drained triaxial compression is shown in Figure 9. It can be observed from Figure 9 that the stress strain (q- ε_a) and the effective stress ratio (q/p') plots obtained from the CD tests are affected by the curing time in a similar way to that in the CU tests (see Fig. 6). Figures. 9(a) and 9(b) demonstrate that the peak stress, q_p and the peak effective stress ratio, (q/p')_p increase after 7 curing days but decrease after 28 curing days. This suggests that the efficiency of lime for improving the engineering properties of organic clay reduces in a long term. Despite experiencing a decrease in the q_p and (q/p')_p at 28 days of curing, the properties after curing for 7 and 28 days were still higher than those of the untreated samples (0 days and 0% lime content). This, in turn, demonstrates that the lime stabilisation of the organic clay containing 1.5% humic acid was successful. However, since the

improvement in the behaviour of clay reduces from 7 to 28 days, the effectiveness of the lime stabilisation appears uncertain over longer curing periods and raises a question about practical viability of the lime stabilisation process in organic clay, particularly in cases where long-term stability is imperative.

The volumetric strain behaviour obtained from the CD tests on lime-stabilised organic clay at different curing periods is presented in Figure 9(c). It can be observed from Figure 9(c) that the largest compression was measured during shearing of the untreated specimen (i.e. 0 curing days). The behaviour of the lime-stabilised clay after 7 days was least compressive. The ε_v - ε_a curve of the specimen sheared after 28 days of curing was in between of those cured for 0 and 7 days. The results presented in Figure 9(c) confirm that the effectiveness of lime stabilisation of organic clay may reduce in a long term as a larger volumetric compression is typically associated with the lower shear strength.



Figure 7 Effect of humic acid content on the drained behaviour of lime-treated organic clay at 7 curing days: (a) q- ε_a , (b) (q/p')- ε_a , (c) ε_v - ε_a



Figure 8 Effect of humic acid content on the effective friction angle of lime-treated specimens sheared after 7 curing days under drained conditions



4.2.3 Comparison of Drained and Undrained Behaviours

It has been widely reported that after 28 days pozzolanic reactions begin to occur (Mohd Yunus 2012). Thus, in this study the CU and CD triaxial test results obtained for samples cured for 28 days were analysed and compared in more detail.

The CU and CD test results obtained for organic specimens prepared at the OLC = 5% and tested at effective confining stresses (σ'_3) of 50 kPa and 100 kPa are presented in Fig. 10 in the form of effective stress path diagrams (also called 'modified' Mohr-Coulomb diagrams).



Figure 9 Effect of curing time on the drained behaviour of limetreated clay with 1.5% humic acid: (a) q- ε_a ; (b) (q/p)'- ε_a ; (c) Δu - ε_a

Figure 10 Effective stress paths and Mohr-Coulomb failure envelopes of lime-treated organic clay: (a) 0%; (b) 0.5%; (c) 1.5%; and (d) 3% humic acid

'Best-fit' failure lines were determined for organic clay specimens with 0, 0.5, 1.5, and 3% humic acid, as shown in Figure 10(a) to 10(d), respectively. Figure 10 shows a good agreement between failure states obtained from CU and CD tests. This means that the effective Mohr-Coulomb failure parameters (i.e. φ' and c') for the organic clay tested in this study could be obtained from either CU or CD tests. The equation of the failure line determined for specimens with different humic acid content are shown in Figure 10. Based on the effective failure envelopes plotted in Fig. 10, the Mohr-Coulomb strength parameters (c' and φ') of the lime-treated organic clay (5% lime) were determined and plotted in Figure 11 versus humic acid content. The Mohr-Coulomb strength parameters determined for untreated (i.e. 0% lime) specimens (Mohd Yunus 2012) are also plotted in Figure 10.



Figure 11 Effect of humic acid on the Mohr-Coulomb strength parameters: (a) friction angle, (b) cohesion intercept

The effect of humic acid content on the peak effective friction angle, φ_p ', is shown in Figure 11(a). It can be seen that the effect of the humic acid content on the φ_p ', is not significant for both limetreated and untreated specimens if the Mohr-Coulomb failure criterion is considered. As shown in Fig. 11(a), φ'_p of the limetreated specimens decreased from 22.3° to 20.9, 20.7, and 18.7° when the humic acid content increased from 0% to 0.5, 1.5, and 3%, respectively. Furthermore, it can be seen from Figure 11(a) that the φ'_p was almost constant when humic acid content was increased from 0.5% to 1.5%. The most significant change in φ'_p was observed beyond 1.5% humic acid content was increased from 20.7° to 18.7° when the humic acid content was increased from 1.5% to 3.0%.

The effect of humic acid content on the φ'_p of untreated specimens was even smaller compared to that of the lime-treated specimens (Mohd Yunus 2012). It can be observed from Figure 11(a) that the φ'_p was equal to 9.6°, 9.2°, 8.6° and 8.5° for the specimens with 0%, 0.5%, 1.5% and 3.0% humic acid content, respectively. Nonetheless, Fig. 11(a) clearly shows that φ'_p of the organic clay tested in this study increased significantly after lime stabilisation regardless of the humic acid content. Thus, the lime stabilisation of the organic clay was efficient.

The effect of the humic acid content on the cohesion intercept (c') is shown in Fig. 11(b). It can be seen that the c' values varied

significantly for both untreated and lime-treated specimens with different humic acid content. For instance, the c' of lime-treated specimens reduced from 24 kPa to 17 kPa, 7 kPa and 5 kPa when the humic acid content increased from 0% to 0.5, 1.5, and 3.0%, respectively. Similar changes in c' were measured for the untreated specimens with different humic acid contents, as shown in Fig. 11(b). However, in contrast to the effect of lime on φ'_p values, the untreated specimens were characterised by higher cohesion compared to that of the lime-treated specimens. This suggests that the effect of lime on the effective friction angle is more significant than that on the cohesion intercept. The shear strength of the lime-treated organic clay determined from the Mohr-Coulomb failure criterion (from Eq. (1) below) is higher than that of the untreated specimens.

$$\tau_{\rm f} = c_{\rm f}' + \sigma_{\rm f}' \tan \varphi_{\rm f}' \tag{1}$$

where τ_f is the shear strength, c_f' is the cohesion intercept, σ_f' is the effective stress at failure, and ϕ_f' is the effective friction angle.

For example, the shear strength of the lime-treated clay sheared at σ'_3 of 50 kPa was 44.1 kPa, 36.1 kPa, 26.0 kPa and 22.0 kPa when the humic acid content as equal to 0%, 0.5%, 1.5% and 3.0%, respectively. Similarly, the τ_f of the untreated specimens was 36.5 kPa, 32.0 kPa, 22.7 kPa and 20.2 kPa for 0%, 0.5%, 1.5% and 3.0% humic acid content, respectively. These values demonstrate that the lime stabilisation is efficient for improving shear strength of organic clay. However, humic acid content has detrimental effects on the shear strength of both untreated and lime-treated organic clay, in particular beyond 1.5% content.

5. CONCLUSIONS

The effects of humic acid content on the engineering properties of lime-treated organic clay were investigated in this paper. Based on the results obtained from laboratory tests, the following conclusions can be drawn:

- Unconfined Compressive Strength (UCS) test results showed that the modification process in lime-treated organic clay reduces when the lime content exceeds 5%. For this reason, 5% was assumed as the optimum lime content (OLC) for the organic clay tested in this study.
- 2) The UCS test results also demonstrated that the undrained shear strength of organic clay increases significantly as early as 7 days after stabilising with lime. However, at longer curing periods the undrained shear strength of limetreated organic clay comprising 0.5%, 1.5% and 3.0% humic acid kept decreasing.Therefore, the lime stabilisation of organic clay cannot be considered successful in the long term even though the undrained shear strengths of lime-treated specimens after 90 days was higher than that of untreated specimens.
- 3) The reduction in pH value at longer curing periods was the main reason for impedance in the improvement of limetreated organic clay in the long term. The pH values of the organic clay decreased with the increasing curing period and fall below 12.40, which is considered to be the minimum value necessary for production of cementing materials during lime stabilisation process. Therefore, the long-term shear strength of lime-treated organic clay reduced indicating that the lime stabilisation of organic clay containing humic acid is not very effective.
- 4) The values of c' and φ'_p determined from CU and DC triaxial compression tests indicate that the effective shear strength of lime-treated organic clay decreases with increasing humic acid content. Overall, a slight loss in strength was recorded for organic clay with 0.5% humic

acid, while a substantial loss in strength was recorded where humic acid content was equal to or greater than 1.5%. These findings revealed that lime may not be the best stabiliser for improving engineering properties of organic clay with more than 1.5% humic acid content. Furthermore, since the shear strength of lime-treated organic clay was higher than that of untreated organic clay, the longterm improvement of the clay was found unsatisfactory. Therefore, other additives (e.g. chloride salts) should be considered in order to improve the effectiveness of stabilisation process of organic clay with high humic acid content.

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