

Effect of Humic Acid on Physical and Engineering Properties of Lime-Treated Organic Clay

N. Z. Mohd Yunus, D. Wanatowski, and L. R. Stace

Abstract—The present work deals with the stabilisation of organic clay using hydrated lime. Artificial organic clays were prepared by adding kaolin and different humic acid contents. Results given by physical testing show that the presence of humic acid has a drawback effect on the untreated organic clay. The decrease in specific gravity value was accompanied by a decrease in dry density and plasticity of clay at higher humic acid contents. Significant increase in shear strength at 7 days of curing period is observed in the lime-treated samples up to 5% lime content. However shear strength of lime-treated organic clay decreases at longer curing periods. The results given by laboratory testing is further verified by microstructure analysis. Based on the results obtained in this study, it can be concluded that the presence of more than 1.5% humic acid reduces significantly the efficiency of lime stabilization in organic clays.

Keywords—Humic acid, kaolin, lime, organic clay

I. INTRODUCTION

ORGANIC clay is defined as a clay with sufficient organic content to influence the soil properties [1]. Organic clays are normally characterized by high plasticity, high water content, high compressibility, low permeability and low shear strength. These characteristics make organic clays unsuitable for engineering construction unless a suitable improvement method is applied.

Lime stabilization is one of the most popular methods of ground improvement. However, stabilization mechanisms in lime-treated organic clay are thought to be affected by high water content and high organic matter content. This is because organic matters have high water holding capacity that limits the water available for the hydration process taking place during stabilization [2]. In addition, high water content may induce more spacing between aggregations, hence reducing a required cementation bonding. Some organic compounds delay or even inhibit the hydration process of lime as they have the tendency to coat the soil particles. Besides, it is believed that organic matter has a strong chemical affinity to calcium, thus when reacting with $\text{Ca}(\text{OH})_2$ it will form insoluble reaction products precipitating out on the clay particles [2]. Organic matters have also a tendency to lower

N. Z. Mohd Yunus is with the Faculty of Engineering, University of Nottingham, University Park, NG7 2RD, United Kingdom, on leave from Universiti Teknologi Malaysia (phone: +44-115-95-18098, fax: +44-115-95-13898, e-mail: evxnmz@nottingham.ac.uk).

D. Wanatowski is with the Faculty of Engineering, University of Nottingham, University Park, NG7 2RD, United Kingdom (e-mail: dariusz.wanatowski@nottingham.ac.uk).

L. R. Stace is with the Faculty of Engineering, University of Nottingham, University Park, NG7 2RD, United Kingdom (e-mail: rod.stace@nottingham.ac.uk).

pH to the point where dissolution of clay minerals no longer takes place [3], [4], [5]. For example, it was reported by [4] that a lime-treated subgrade with pH of 10 was unsuitable for construction even though a higher percentage of lime was added. This is because the clay particles failed to react with lime to form a cementing product during the pozzolanic reaction [4].

There are two main fractions of organic matter present in organic soils, namely humic acid and fulvic acid. Based on several studies carried out by previous researchers, it was agreed that humic acid is the main constituent of organic matter affecting the strength development of stabilized soil [6], [7], [8]. It was also reported that the shear strength of lime-treated organic clay is affected by the presence of more than 1% humic acid in soil [9], [10]. However, no clear evidence has been published to support this hypothesis. For instance, [11] showed that soft clay with high organic matter content of 14% was successfully stabilized with 7% of lime. However, [9] reported that clay with 12.8% organic matter content exhibited only a slight increase in shear strength after being stabilized with 10% of lime. These results suggest that the effects of organic matter, especially humic acid, on the strength development of organic clays are not fully understood. Further study is still required to quantify the effects of humic acid on physical and engineering properties of lime-stabilized organic clay.

This paper presents results of experimental study carried out on artificial organic clay with different humic acid contents. The effects of humic acid on selected physical and engineering properties of organic clay are investigated and summarized. The results of laboratory tests are discussed in light of microstructure analysis carried out on selected clay specimen by using the Scanning Electron Microscope (SEM).

II. MATERIALS

The soil used in this research was an artificial organic clay. The soil samples were prepared by mixing commercial kaolin with different percentages of humic acid (0%, 0.5%, 1.5% and 3%) according to dry mass of kaolin.

Table I summarizes chemical composition of organic clay with different humic acid contents. In general, the presence of humic acid can be found from the occurrence of carbon and oxide ions based on chemical element analysis. It can be seen from Table I that the total amounts of carbon and oxide ions increase with increasing humic acid content. In addition, the presence of humic acid can be identified by the increase in total amount of sulphur and phosphorus. As a result, the amount of pozzolan minerals such as silica and alumina decrease with an increase of humic acid content.

TABLE I
CHEMICAL COMPOSITION OF TESTED SOILS

Element	Wt%		
	K0.5HA	K1.5HA	K3.0HA
C	2.8	2.74	20.16
O	50.45	54.54	48.97
Na	0.36	0.41	0.81
Mg	0.65	0.64	0.56
Al	16.94	18.47	12.8
Si	24.74	21.52	14.07
P	0.18	0.28	0.16
S	0.05	0.12	0.42
Cl	0	0.01	0.19
K	3.42	0.79	0.59
Ca	0.02	0.04	0.33
Ti	0	0.02	0.18
V	0	0.01	0.01
Fe	0.39	0.4	0.74

Note: K=Kaolin, HA=humic acid

III. SPECIMEN PREPARATION AND TESTING PROGRAM

All the soil specimens tested in this study were prepared using standard procedures described in the ASTM standard [12]. As recommended by the standard [12], before mixing, the specimens were oven-dried at 60°C until the constant weight was obtained. All the specimens of untreated clay tested in this study were 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 kaolin-humic 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. All the tests were carried out according to the British Standard [13].

The results obtained from the compaction tests were used to prepare specimens for strength testing. Each of the specimens of lime-treated organic clay was prepared according to the maximum dry density (MDD) and optimum moisture content (OMC) of the untreated clay with corresponding humic acid content. By knowing the volume of mould and the MDD, the required dry mass of soil was calculated. The appropriate quantity of hydrated lime was then calculated based on the dry mass of the untreated soil. Then, these amounts of dry soil and dry lime were mixed with water according to the OMC. 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 the mould at a 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 cling film to preserve the water content and to keep them free from carbon dioxide (CO₂). The specimens were then cured in desiccators at 20°C and with humidity more than 90% for 7, 28, and 90 days, respectively. For each soil type, two duplicate specimens were tested to ensure repeatability of test results. The specimens were 76 mm in height and 38 mm in diameter.

IV. RESULTS AND DISCUSSIONS

The investigation of the effects of humic acid content was first performed on untreated sample (i.e., artificial organic clay) to obtain benchmark data for comparison with lime-treated specimens. A preliminary testing on the artificial organic clay was carried out to evaluate the suitability of this soil as a research material. Firstly, plastic and liquid limits, specific gravity, pH and compaction characteristics were determined [13]. After that, the unconfined compressive strength (UCS) tests were carried out to identify the effect of humic acid content on the engineering properties of soils. Finally, the effect of humic acid content on lime-treated organic clay was determined by the UCS test at different curing periods.

A. Physical Properties

The physical properties determined for the untreated artificial organic clay are summarized in Table II. It can be seen that the specific gravity of soil prepared with humic acid is low when compared to inorganic clay (K in Table II). It was also found that with a higher percentage of humic acid a lower density of soil was obtained, which is similar to what has been reported by other researchers [7], [8], [9], [14], [15].

TABLE II
PHYSICAL PROPERTIES OF UNTREATED ORGANIC CLAY

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	31.4	29.5	26.5
Specific gravity	2.61	2.53	2.51	2.47
pH	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.

Fig. 1 shows the range of plasticity determined for organic clay with different humic acid contents. The upper and lower boundaries were determined by the plastic and liquid limit values, respectively. The difference between the liquid and plastic limits defines the plasticity index (I_p).

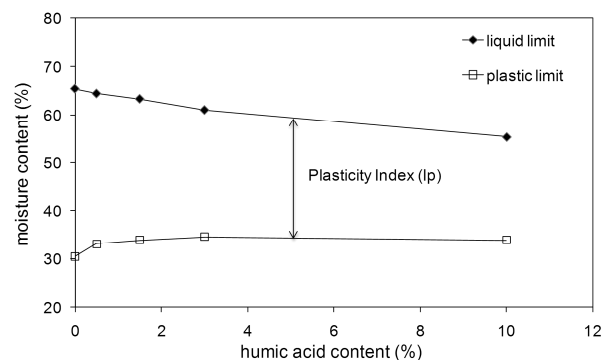


Fig. 1 Effect of humic acid content on plasticity of organic clay

As shown in Fig. 1, the liquid limit of clay decreased with increasing humic acid content. On the other hand, the plastic

limit increased with increasing humic acid content. Consequently, the plasticity index (I_p) kept reducing with increasing humic acid content, which agreed well with experiments carried out by other researchers [3], [15].

The results shown in Fig. 1 also indicate that all the organic clay specimens are suitable for lime stabilization according to the ASTM, as their plasticity indices are larger than 10.

The results presented in Fig. 1 are very important from practical point of view because the shear strength and stiffness of clays are related to the plasticity. The greater the I_p , the more plastic behaviour of soil will be obtained. For example, the liquid limit of soil is defined as the minimum moisture content at which a soil begins to flow when a very small shearing force is applied. Beyond this limit, soil may undergo permanent deformation by its own weight or under application of a very small stress. In other words, the soil will behave like a liquid.

As mentioned earlier, the standard compaction tests (so-called Proctor tests) were carried out on untreated clay specimen. The results obtained from the compaction tests are plotted in the form of compaction curves in Fig. 2. It can be seen from Fig. 2 that the maximum dry density (MDD) of the untreated organic clay decreased with increasing humic acid content. At the same time, the optimum moisture content (OMC) increased at higher humic acid contents. Similar trends in compaction behaviour have also been observed by other researchers [3], [15], [16]. It should also be mentioned that higher dry density of soil is normally associated with higher shear strength. Therefore, based on the compaction curves shown in Fig. 2, it is expected that the strength of organic clay prepared with the higher percentage of humic acid will be significantly lower compared to inorganic clay (i.e., with 0% humic acid).

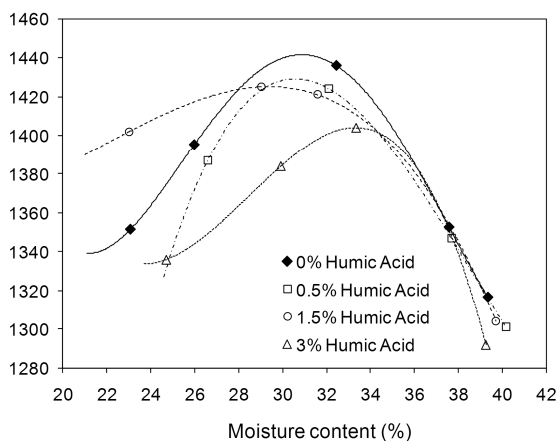


Fig. 2 Effect of humic acid content on compaction characteristics of organic clay

B. Shear Strength

The Unconfined Compressive Strength (UCS) tests were first carried out on the specimens of untreated organic clay with different humic acid contents. Although no curing was

required for the untreated organic clay, the specimens with different humic acid contents were tested after 7, 28 and 90 days to ensure consistency with the results obtained from tests on the lime-treated organic clay. Nonetheless, it was found that the strengths of untreated clay were not affected by the curing time. Therefore, average values of the strength obtained from specimens with different humic acid contents were used for further analysis.

Fig. 3 shows the relationship between the average undrained shear strength of clay and the humic acid content. It can be seen from Fig. 3 that the shear strength of organic clay decreases with increasing humic acid content. For instance, the organic clay with 3% humic acid content experienced the strength loss of about 44.6% compared to inorganic clay (i.e., with 0% humic acid content).

The shear strength test results plotted in Fig. 3 are consistent with those obtained from the compaction tests, which showed a decrease in the dry density with increasing humic acid content (see Fig. 2).

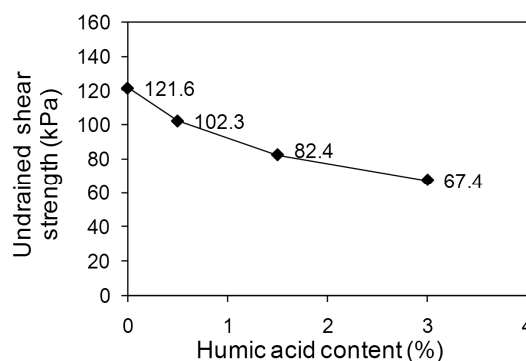


Fig. 3 Effect of humic acid content on the shear strength of organic clay

The UCS tests were also carried out on lime-treated organic clay. However, before the UCS tests, a suitable amount of lime added to organic clay had to be determined. This was essential to ensure that the amount of lime added to organic clay is sufficient for the occurrence of modification and stabilization processes.

The initial consumption of lime (ICL) test was conducted as a first step to identify the minimum amount of lime required for stabilisation process to take place. The ICL is defined as the minimum amount of lime in a treated-clay required to give a pH of 12.40 [17]. The ICL gives an indication of the minimum amount of lime that is required to be added to a material to achieve a significant change in its properties.

Fig. 4 shows the relationship between pH values and lime content in the lime-treated organic clays. It can be seen from Fig. 4 that approximately 2% of lime was needed to achieve pH of 12.4. However, more than 2% of lime is normally required to ensure a long term strength gain achieved from the lime stabilization process [3], [6], [10], [15]. Therefore, 5% was chosen as the minimum lime content used for lime stabilization of organic clay. In addition, organic clay was

stabilized with 8%, 10% and 15% of lime to analyze the effect of lime content on the shear strength of lime-treated organic clay.

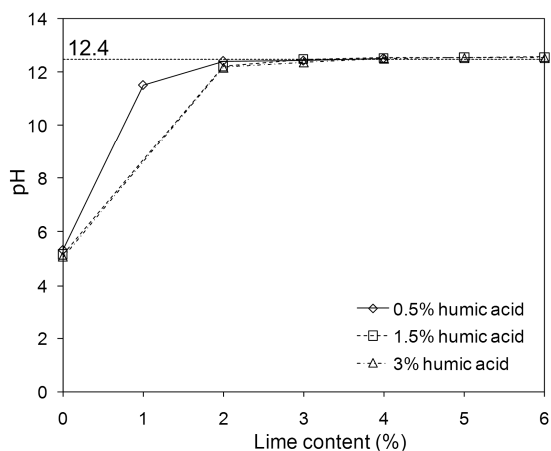


Fig. 4 Relationship between pH value and lime content in lime-treated clay

The results of the UCS tests carried out on lime-treated organic clay with different humic acid contents (0%, 0.5%, 1.5%, 3%) and lime contents (5%, 8%, 10%, 10%) are shown in Fig. 5. All the specimens were cured for 7 days.

It can be seen from Fig. 5 that the undrained shear strength of all the specimens with 5% lime content increased significantly compared to the strength of untreated clay (i.e., 0% lime). However, the shear strength of specimens with higher than 5% lime contents reduced gradually with increasing lime content regardless of the humic acid content in the clay. Therefore, 5% was considered as the optimum lime content (OLC) for each type of organic clay.

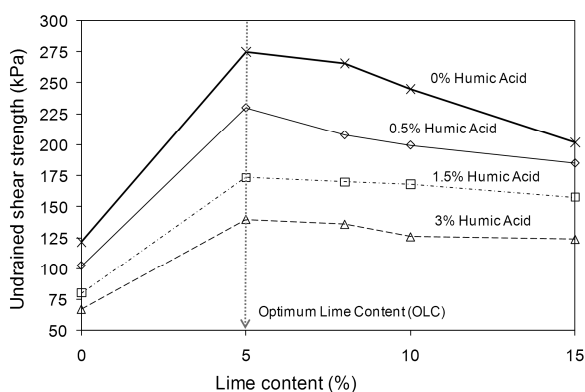


Fig. 5 Effect of lime content on the shear strength of clay after 7 days of curing

Fig. 5 also shows that the shear strength of organic clay decreases with increasing humic acid content which proves that the presence of humic acid in organic clay diminishes the shear strength. Similar results were also reported by other researchers [4], [5], [7].

The effect of curing time on the shear strength of lime-treated organic clay was also investigated in this study. The results of the UCS tests carried out on organic clay stabilized with 5% of lime, after 7, 28, and 90 days of curing, are shown in Fig. 6.

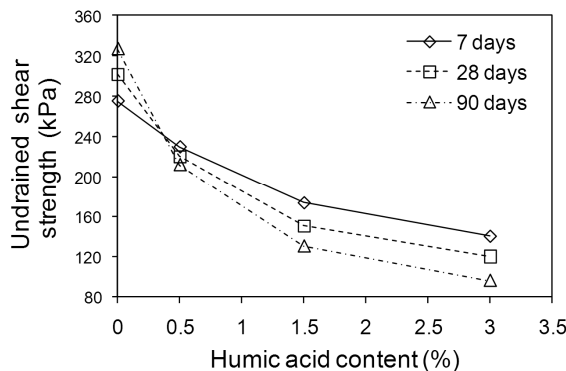


Fig. 6 Effect of curing period on the shear strength of lime-treated organic clay

It can be seen from Fig. 6 that the undrained shear strength of organic clay reduced with increasing curing period. For example, the strength of the organic clay with 3% of humic acid reduced from 120 kPa at 28 days to 96 kPa at 90 days. The experimental results plotted in Fig. 6 also show that the strength losses from 7 to 28 days for kaolin prepared with 0.5%, 1.5% and 3% humic acid were about 4.1%, 13.4% and 14.4%, respectively. Meanwhile, the total strength losses of the same clays from 7 to 90 days were 7.8%, 25.1% and 31.7%, respectively. Percentages of strength loss increased with increasing humic acid content and with curing period.

The results shown in Fig. 6 clearly demonstrate that lime stabilization of organic clay with high humic acid content is not very efficient in a long term. Similar observations were reported by other researchers [5], [9], [10]. This behaviour is different from that observed for lime-stabilized inorganic clays, where the shear strength increases with curing time [18], [19].

C. Microstructure Analysis

Microstructure of clay specimens sheared in the UCS tests was analyzed by the scanning electron microscope (SEM). SEM enables the surface of clay samples to be imaged by scanning it with a high-energy beam of electrons. This information is produced from the signals given by the electrons when reacting with the atoms.

Figs. 7(a) and 7(b) show SEM images of untreated inorganic clay (i.e., 0% humic acid content) and of organic clay with 0.5% humic acid content, respectively. Both images were taken at 50000x magnification. It can be seen from Fig. 7 that both specimens have similar microstructures. Plate-like and hexagonal individual particles were observed in both clay specimens. Similar observation was reported by [19] who found that a low humic acid content does not affect significantly the microstructure of clay. The fabric of both

specimens shown in Fig. 7 also reveals no apparent aggregations, which confirms that no chemical reactions were involved in untreated specimens.

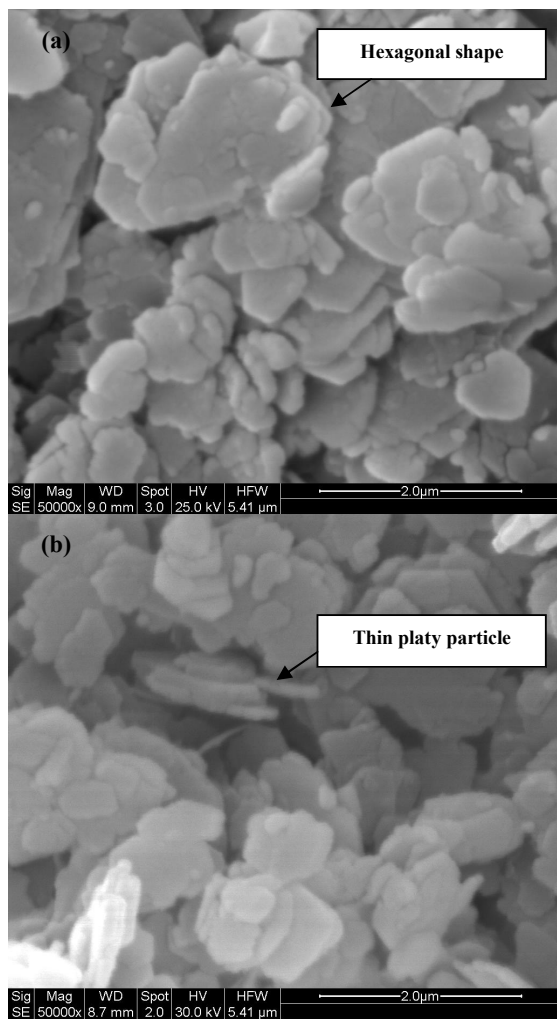


Fig. 7 SEM micrographs of untreated clay: (a) 0% humic acid, (b) 0.5% humic acid

In contrast to organic clay with lower humic acid contents, some cracks could be observed on clay particles at 1.5% humic acid content (Fig. 8a). These cracks became more noticeable in clay with 3% humic acid content (Fig. 8b). The occurrence of cracks on individual particles suggests that the breakage of particles took place due to obstruction of humic acid. The effect of humic acid on microstructure of clay particles was most pronounced in specimens with 3% humic acid content. It can be seen from Fig. 8(b) that some particles started to crumble.

The SEM micrographs of specimens with 0.5% and 3% humic acid treated with 5% of lime and cured for 28 days are shown in Fig. 9. The duration of 28 days curing period represents stabilization process. Therefore, it is expected that cementing products such as Calcium Silicate Hydrate (CSH) and Calcium Aluminate Hydrate (CAH) are fully formed in the soil structure.

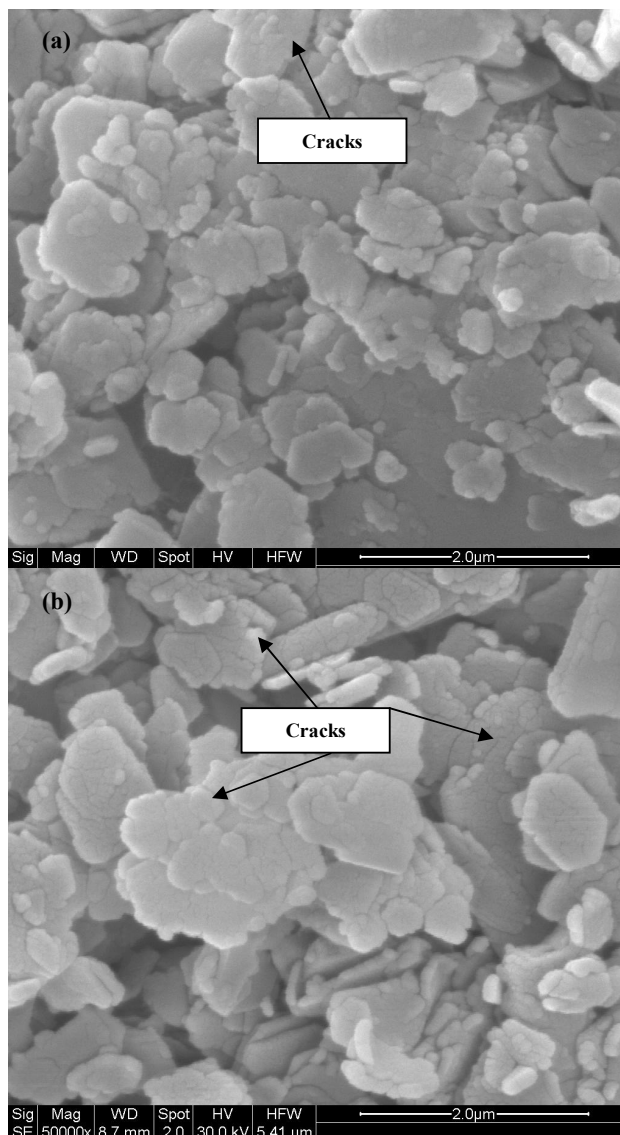


Fig. 8 SEM micrographs of untreated clay: (a) 1.5% humic acid, (b) 3% humic acid

The SEM images of lime-treated organic clay revealed the appearance of well aggregated structure where particles clump to each other to form bigger units, as shown in Fig. 9. It can also be observed that less pronounced individual plates are observed in lime-treated clay (Fig. 9) compared to untreated soil (see Figs 7 and 8). The crack appearance in untreated clay with 3% humic acid was also reduced after being treated with 5% lime. On the other hand, specimens with 3% humic acid showed more voids between aggregates compared to the lime-treated organic clay with 0.5% humic acid. This suggests a fewer cementing products filling the pores of lime-treated organic clay with higher humic acid content. This explains why the shear strength of organic clay reduces with increasing humic acid content.

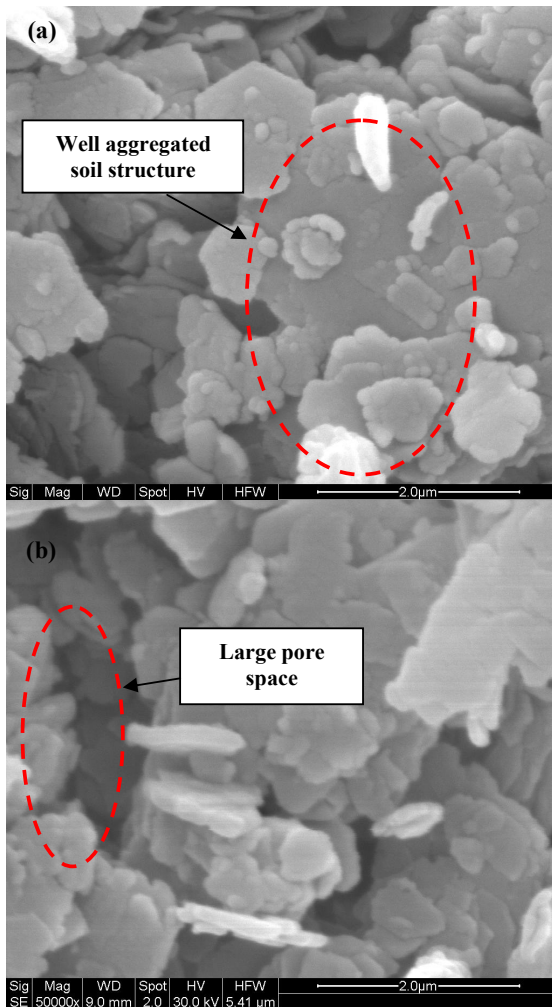


Fig. 9 The structure of clay after treatment with 5% lime: (a) 0.5% humic acid content, (b) 3% humic acid content

V. CONCLUSIONS

In this paper, the effects of humic acid on physical and engineering properties of lime-treated organic clay were investigated in this study. Based on the experimental results obtained in the study the following conclusions can be made.

- 1) The liquid limit of organic clay decreased with increasing humic acid content. On the other hand, the plastic limit increased with increasing humic acid content. Nonetheless, all the specimens of organic clay with various amounts of humic acid were suitable for lime stabilization. This is because the plasticity index (I_p) of each type of organic clay was higher than 10.
- 2) The shear strength of lime-treated organic clay reduced when the lime content exceeded 5%. Thus, 5% of lime was identified as the optimum lime content (OLC) for the organic clay tested in this study.
- 3) The shear strength of lime-treated organic clay reduced at longer curing periods. The total strength loss determined between 7 and 90 days of curing was 7.8%, 25.1% and 31.7%, for clay with 0.5%, 1.5%, and 3% humic acid,

respectively. These results show that the presence of at least 1.5% humic acid in organic clay reduces significantly the efficiency of lime stabilisation process.

- 4) The SEM images of lime-treated organic clay revealed the appearance of well aggregated microstructure created during stabilization process. However, significantly more voids between aggregates were detected in clay with higher humic acid contents. This explains why the shear strength of lime-treated organic clay reduces with increasing humic acid content.

REFERENCES

- [1] ASTM, Standard D2487, "Standard Practice for Classification of Soils for Engineering Purposes (Unified Soil Classification System)," *Annual Book of ASTM Standards*, Vol. 04.08, ASTM International, West Conshohocken, PA, pp. 249-260, 2004.
- [2] H. Chen, and Q. Wang, "The behaviour of organic matter in the process of soft soil stabilization using cement," *Bulletin Eng. Geol. Env.*, vol. 65, no. 4, pp. 445-448, 2006.
- [3] B. Huat, S. Maail, and T. Ahmed Mohamed, "Effect of chemical admixtures on the engineering properties of tropical peat soils," *American J. App. Sciences*, vol. 2, no. 7, pp. 1113-1120, 2005.
- [4] D.-H. Chen, Z. Si, and M. Saribudak, "Roadway Heaving Caused by High Organic Matter," *J. Perf. Const. Facilities*, vol. 23, no. 2, pp. 100-108, 2009.
- [5] W. Zhu, C. F. Chiu, C. L. Zhang, and K. L. Zeng, (2009). "Effect of humic acid on the behaviour of solidified dredged material," *Can. Geot. J.*, vol. 46, no. 9, pp. 1093-1099, 2009.
- [6] K. E. Clare, and P. T. Sherwood, "Further studies on the effect of organic matter on the setting of soil-cement mixtures," *J. App. Chem.*, vol. 6, no. 8, pp. 317-324, 1956.
- [7] K. Onitsuka, C. Modmoltin, M. Kouno, and T. Negami (2002). "The effect of humic acid on lime stabilized Ariake Clay," in *Proc. 12th Int. Offshore and Polar Eng. Conf.*, Kitakyushu, Japan, pp. 577-583, 2002.
- [8] K. Onitsuka, C. Modmoltin, M. Kouno, and T. Negami, "Effect of organic matter on lime and cement stabilized Ariake clays," *Proc. Jap. Soc. Civ. Eng.*, No. 729, pp. 1-13, 2003.
- [9] S. Koslanant, K. Onitsuka, and T. Negami, (2006). "Influence of salt additive in lime stabilization of organic clay," *Geot. Eng. J.*, vol. 37, pp. 95-101, 2006.
- [10] P. Harris, O. Harvey, S. Sebesta, S. R. Chikyala, A. Puppala, and S. Saride, "Mitigating the effects of organics in stabilized soil," Technical Report No. 0-5540-1, Texas Transportation Institute, USA, 2009.
- [11] M. A. Sakr, and M. A. Shahin, "Utilization of lime for stabilizing soft clay soil of high organic content," *Geot. Geol. Eng.*, vol. 27, pp. 105-113, 2009.
- [12] ASTM, Standard D5102-96, "Standard Test Method for Unconfined Compressive Strength of Compacted Soil-Lime Mixtures," *Annual Book of ASTM Standards*, Vol. 04.08, ASTM International, West Conshohocken, PA, pp. 1073-1078, 2004.
- [13] British Standard 1377, "Methods of test for soils for civil engineering purposes," British Standard Institution, London, 1990.
- [14] H. Ahnberg, "Effects of back pressure and strain rate used in triaxial testing of stabilized organic soils and clays," *Geot. Test. J.*, vol. 27, no. 3, pp. 250-259, 2004.
- [15] A. J. Puppala, S. P. Pokala, N. Intharasomba, and R. Williammee, "Effects of organic matter on physical, strength, and volume change properties of compost amended expansive clay," *J. Geot. Geoen. Eng.*, vol. 133, no. 11, pp. 1449-1461, 2007.
- [16] H. Ahnberg, S.-E. Johansson, H. Pihl, and T. Carlsson, "Stabilising effects of different binders in some Swedish soils," *Ground Improvement*, vol. 7, no. 1, pp. 9-23, 2003.
- [17] British Standard 1924-2, "Stabilized materials for civil engineering purposes. Part 2: Methods of test for cement-stabilized and lime-stabilized materials," British Standard Institution, London, 1990.
- [18] F.G. Bell, "Lime stabilization of clay minerals and soils," *Eng. Geol.*, vol. 42, no. 4, pp. 223-237, 1996.
- [19] R. James, A. H. M. Kamruzzaman, A. Haque, and A. Wilkinson, (2008). "Behaviour of lime-slag-treated clay," *Ground Improvement*, vol. 161, no. 4, pp. 207-216, 2008.