Stress-strain characteristics of two natural soils subjected to long-term acidic contamination

Ivan Gratchev[1] and Ikuo Towhata[2]

1) Lecturer, Griffith School of Engineering, Centre for Infrastructure Engineering and Management, Griffith University, Australia. *Email: ivangratchev@gmail.com*

2) Professor, Department of Civil Engineering, The University of Tokyo, Japan.

Abstract. This paper seeks to investigate the effect of acidic contamination on the stress-strain characteristics of two natural soils with different mineral composition: Kawasaki mud that contains montmorillonite, and Yurakucho silt that has kaolinite as the dominant clay mineral. To reproduce the process of soil contamination in the laboratory, a special container was designed in which soil samples were leached with solutions of sulfuric acid for a period of almost 300 days. Laboratory investigation included a series of undrained triaxial compression tests on the leached samples. The obtained results indicated that acidic contamination had a strong influence on the strength characteristics of soils, and the mineralogy of the clay fraction as well as the concentration of acid in the pore fluid could significantly influence the stress-strain behavior of studied soils. It was found that for the Kawasaki mud, a decrease in pH from 7.3 to 6.0 led to an increase in the strength of soil. For the Yurakucho soil, a small decrease in pH (pH=6.0) also slightly increased the strength of soil. However, further decrease in pH (pH=4.0) caused a significant reduction of the strength. An attempt was made to provide a rational explanation for the observed behavior of soils based on the fundamentals of colloid chemistry and available literature.

KEY WORDS: acid; contamination; strength; buffer capacity; clay mineralogy; undrained triaxial tests.

1. Introduction

In the past few decades, a great deal of research has been conducted to understand the influence of pore water chemistry on the strength characteristics of fine-grained soils. A few examples of such studies are: Sridharan and Prakash (1999), Anandarajah and Zhao (2000), Ratnaweera and Meegoda (2006), Sunil et al. (2009), Man and Graham (2010) who carried out a number of laboratory tests to understand the influence of chemicals on the stress-strain behavior of soil, as well as Mesri and Olson (1970), Di Maio and Fenelli (1994), and Tiwari et al. (2005) who investigated the influence of pore fluids on residual shear strength.

The aforementioned studies significantly improved our understanding of the effects of pore fluids on the geotechnical properties of fine-grained soils, and provided useful insights into the mechanisms of soil-water-chemical interaction. However, most of the research has focused on organic liquids or inorganic fluids such as sodium chloride, while little has been done to understand the effects of pH, especially acidic water, despite the fact that acid contamination has become an increasing concern around the world. The available literature indicates that acidic contamination of soil can occur through natural processes such as weathering of pyrite in mudstone (Chigira, 1990), or due to anthropogenic activities such as municipal waste storage, accidental spills or acidic rains (Kamon et al., 1997). Only recently has this problem been recognized, and a few studies have been performed to investigate the effects of acid on soil properties such as compressibility (Gratchev and Towhata, 2011), residual shear strength (Gajo and Maines, 2007) and cyclic shear strength (Gratchev and Sassa, 2009). Yet the effect of acidic fluids on the strength of soil remains unclear. This study seeks to shed light on this issue by providing laboratory data concerning the stress-strain behavior of two natural soils subjected to acidic contamination.

It is noted that in many studies the contamination of soil was achieved through mixing soil slurry with acidic fluids as it provides experimental data within a reasonable time frame. However, this approach tends to neglect the effect of soil structure on the process of soil-acid-water interaction. For this reason, this study makes an attempt to reproduce the process of soil contamination in the laboratory without remolding soil samples. To achieve this, a special container was designed in which soil samples were leached with solutions of sulfuric acid for several months until the desired degree of contamination was reached.

The Kawasaki mud containing montmorillonite and Yurakucho silt with kaolinite as the dominant clay mineral were selected for this study to understand the effect of clay mineralogy on the geotechnical properties of contaminated soils. Buffer capacity tests were conducted to assess the ability of each soil to neutralize acid. A series of undrained triaxial tests were carried out on the leached samples to study the strength characteristics of soil at different pH conditions. This paper presents and discusses the obtained results.

2. Review of previous works

In recent years, a number of studies have been performed to investigate the effect of pore water chemistry on the strength characteristics of soil. Anson and Hawkins (1998), Moore and Brunsden (1998), and Tiwari et al. (2005) have studied the effect of calcium and sodium chloride on the residual shear strength of weathered mudstone, and reported that high concentrations of ions in the pore water can significantly decrease the diffuse double layer of clay particles, a process that would lead to a greater residual shear strength. Ratnaweera and Meegoda (2006) have researched the influence of organic fluids on stress-strain behavior of soil, and noted that changes in the strength mostly depended on the dielectric constant of the organic fluids. Anandarajah and Zhao (2000) performed a series of tests on kaolinite with various organic fluids, and suggested that the observed increase in the strength of soil was partially due to a decrease in the double-layer repulsive force and the consequent increase in the van der Waals attractive forces acting between the clay particles. Gratchev et al. (2006, 2007) performed studies on the effect of pore fluid chemistry on undrained cyclic behavior of artificial soil mixtures and natural clays, and reported that the cyclic shear strength can be greatly influenced by the type of ions and their concentrations in the pore water.

Although the effect of organic and inorganic pore fluids on the strength of soil seems to be well understood, the influence of acidic pore water on the behavior of soil is still unclear mostly due to the limited number of studies as well as the complexity of processes that may occur in soil during acid-soil interaction. As summarized in Wang and Siu (2006a,b), Gajo and Maines (2007), Gratchev and Sassa (2009), and Gratchev and Towhata (2011), the properties of soil in acidic environment can be affected through several processes, the most important of which are briefly reviewed below.

- Acidic fluids may dissolve the chemical bonding and cementation between clay particles, forming more open clay microfabrics. Imai et al. (2006), and Gratchev and Towhata (2009, 2011) studied the behavior of undisturbed clay samples from the Osaka Bay, Japan, and noted that acid could dissolve the calcium carbonate that served as a cementing agent in this soil. A series of compression tests performed by the investigators revealed that the dissolution of calcium carbonate caused a significant increase in the soil's compressibility.

- At low pH values, when the concentration of H^+-ions greatly increases, a reduction in the diffuse double layer thickness will occur, according to Mitchell (1993), and Van Olphen (1991). This collapse of the diffuse double layer will alter the geotechnical properties of soil such as hydraulic conductivity (D'Appolonia, 1980; Kashir and Yanful, 2001), compressibility (Gajo and Maines, 2007; Gratchev and Towhata, 2009), cyclic shear strength (Gratchev et al., 2007), and plasticity (Gratchev and Sassa, 2009). Gajo and Maines (2007) reported that for bentonite, this process may also lead to an increase in the residual shear strength of soil.

- It has long been recognized (Van Olphen, 1991; Mitchell, 1993) that charges on the broken bonds of clay particles depend on pH environment. They become increasingly positive at low pH values due to the adsorption of H^+ ions, and more negative at high pH values due to the adsorption of OH^- ions. Although this may occur in different clay minerals, this phenomenon has the most pronounced effect in kaolinite (Ma and Eggleton, 1999; Li and Li, 2000; Jozefaciuk, 2002), where it may produce different particle associations. Van Olphen (1991) noted that the edge-to-face (E-F) associations (flocculation) tend to prevail at low pH conditions ($pH<5.5$) while the face-to-face (F-F) associations (dispersions) predominate at high pH conditions ($pH>7.5$). Results of previous studies indicated that kaolinitic soils with an open-flocculated structure typically exhibit greater liquid limit, permeability, and strength (Sridharan et al. 1988; Mitchell, 1993; Sridharan and Prakash, 1999). Anandarajah and Zhao (2000) suggested that the higher strength of the flocculated structure in kaolinite was due to an increase in the net of interparticle attractive forces (van der Waals forces) which will bring some additional strength to the clay structure. However, Wang and Siu (2006b), who studied undrained triaxial behavior of kaolinite with distilled water and acidic fluids, reported that the strength of kaolinite with a flocculated structure at pH=4.0 was significantly smaller than that obtained for the specimen with distilled water (pH=7.8). Based on this finding, Wang and Siu (2006b) concluded that the interparticle attractive forces do not seem to have a strong influence on the undrained strength of kaolinite, and it is "the initial packing density, i.e., denser or looser," that was the dominant factor in determining the strength of soil.

- It should be noted that the aforementioned processes are dominant for the conditions when the pH varies from 3 to 6, which is the range that is commonly encountered in natural systems. Van Olphen (1991), Mitchell (1993) noted that at extremely high concentrations of acid ($pH\approx1$), significant changes in the mineral structure may occur (for example, dissolution of Al^{3+}), a process that may affect the undrained strength of soil (Spagnoli et al., 2012). To avoid such an alteration of the mineral composition, only light concentrations of acid were used in this research.

From the above review, it can be inferred that the degree to which acidic contamination can affect the properties of soil depends greatly on the mineralogy of clay fraction. Laboratory data obtained for montmorillonitic soils indicate that in an acidic environment, high concentrations of H^{+}-ions would suppress the diffuse double layer of clay particles while for kaolinitic soil, a decrease in pH may result in the formation of more open flocculated structures. This paper seeks to investigate how such changes in soil structure can affect the stress-strain behavior of soil.

3. **Experimental program**

3.1. Soils tested and liquids used

Samples of Yurakucho silt were collected from a construction site in the north part of Tokyo in blocks (cubic shape with about 50 cm in size), transported to the geotechnical laboratory of the University of Tokyo, and kept under the constant temperature. A series of tests, including grain size distribution, Atterberg limits and X-ray analysis were performed to determine the index properties as well as clay mineralogy of the soil. The results of Atterberg limits tests conducted on specimens in distilled water showed that the soil had the liquid limit (LL) of 50.9%, and the plasticity index (PI) of 15.8. The fines content (<0.074 mm) was found to be 42%, and the clay fraction (<0.002 mm) around 17%. The X-ray analysis, which was performed by the Analysis Center Co. Ltd, Tokyo, revealed the presence of kaolinite and chlorite.

The Kawasaki mud, a relatively young, non-consolidated deposit with a high water content, was selected for this research due to its mineral composition that included montmorillonite and chlorite. It was originally dredged from the Kawasaki Port in Tokyo Bay and stored in freshwater at the Port and Airport Research Institute (PARI) for several months. The fines content was determined to be about 81% while the clay fraction was 36%. The liquid limit and plasticity index of the Kawasaki mud were 58.4% and 25.6, respectively. According to the Casagrande plasticity chart, this soil can be classified as “silt of high compressibility”. It is believed that due to the origin of the Kawasaki mud some organic matter might be present in the soil samples before they were leached with acidic fluids.

Distilled water (pH=6.9) and solutions of commercially available sulphuric acid

(0.01M H_2SO_4) were used in this study. Sulphuric acid was diluted to the desired concentrations with pH values of 4.0. All pH measurements were performed by a pH meter (accuracy ±0.01).

3.2. *Test procedure*

3.2.1. Buffer Capacity

Buffer capacity was determined experimentally by titrating each clay with increasing concentrations of sulphuric acid (Yong et al., 1990; Gratchev and Towhata, 2011). The acid solutions were first prepared at certain concentrations, and then added to the soil at a ratio of 1:10 for soil:acid solution, using 4 g of dry soil and 40 g of acid solution. The pH of the soil solution was measured after the soil suspension sample was thoroughly shaken and allowed to stand for at least 24 hr.

3.2.2. Specimen Preparation

To reproduce the process of soil contamination by acidic fluids, a few containers as schematically shown in Fig. 1 were utilized in this research. The soil sample was sandwiched between two flat perforated PVC plates, which were 5 mm thick with circular holes of diameter 6 mm at 6 mm spacing. Filter paper was placed in between the sample and the plates to prevent loss of the soil. The soil was kept confined under the pressure of 15 kPa to ensure the same stress conditions during leaching.

The block samples of Yurakucho silt were first cut to prepare several soil specimens with the height of 12 cm and width of 5.5 cm, and then placed in the container. A liquid of pH=4.0 was poured into the container to initiate the interaction between the soil and acidic water. The container was covered with the lid to prevent vaporization of the fluid. In the first few weeks, the measurements of pH were performed on a daily basis to monitor changes in the pH of the liquid prompted by soil-chemicals-water interaction. During this time it was observed that some changes in the pH occurred in the first 3-4 days before its value became constant after approximately 6-7 days. Based on the acquired experience, it was decided that a 7-day time interval was sufficient for the chemical equilibrium between the soil and acidic solution to occur. The same procedure was utilized to leach samples of Kawasaki mud with acidic fluids. The Kawasaki mud at its natural water content was placed in the container and allowed to soak in acidic solutions with pH=4.0 for about 7 days. After 7 days the liquid was removed from the container, and its pH was measured and recorded. The container was then re-filled with the original acidic solution to maintain the desired value of pH throughout the tests. Records of the measured pH values for Yurakucho silt and Kawasaki mud are shown in Fig. 2. When the pH of pore fluids reached the desired values, soil samples were retrieved from the container for a series of undrained triaxial tests.

It is noted that a few reference tests were performed on specimens saturated with distilled water. To prepare these specimens, the same container as shown in Fig. 1 was used; however the soil was allowed to soak in distilled water only for 1 week.

In addition, the Atterberg limits of the tested soils were also measured: liquid limit was obtained by Casagrande's method, a plastic limit by standard procedure. The obtained results for both soils at different pH values are summarized in Table 1.

3.2.3. Triaxial undrained compression tests

The specimens were tested one-by-one in a conventional triaxial apparatus. The specimens of the Yurakucho silt leached with acidic fluids were trimmed to a size of 50 mm in diameter and 100 mm in height, and set up in a triaxial cell. As the Kawasaki mud was a soft, non-consolidated deposit, a different procedure was employed for specimen preparation. The leached samples of Kawasaki mud were first pre-consolidated to an effective vertical stress of 50 kPa using an one-dimensional consolidometer. During this stage, an overburden stress was applied in increments of 12.5, 25 and 50 kPa while the drainage was permeated at the both ends of the sample. Each stress was kept for an average of 3 days to achieve at least 90% of consolidation. Then the soil was retrieved from the consolidometer, trimmed to a size of 50 mm in diameter and 100 mm in height, and placed in a triaxial cell.

For all the tests, strips of filter paper were used around the specimen to increase the rate of pore pressure equilibration during undrained tests. After the specimen was placed in the triaxial apparatus it was saturated with the same liquid as the one that was used for leaching to ensure the same chemical conditions throughout the leaching and testing stages. The back pressure was used to ensure a B-value of at least 97%. All specimens were normally consolidated to either of the following isotropic pressure values (σ_3'): 50, 100, or 150 kPa. Following the Japanese Standard for undrained triaxial compression tests (JGS 0523-2000), the specimen was loaded axially with a constant axial strain rate of 0.05%/min to ensure uniform distribution of pore water pressure in the specimen. The experiment was terminated when the axial strain reached a value of 15%.

For the Yurakucho soil, three series of tests were performed on specimens saturated with distilled water (1), leached with acidic fluids to a pH of 6.0 (2), and pH of 4.0 (3). Due to the limited amount of Kawasaki mud available for this research, only specimens with distilled water and specimens leached to a pH value of 6.0 were tested.

4. Results and Discussion

4.1. Buffer Capacity

The titration curves of pH against sulfuric acid input for the two clay suspensions are shown in Fig. 3. Also included in the figure is the titration curve of a blank (a solution of sulfuric acid in the absence of soil). When mixed with distilled water, the Yurakucho silt suspension has a slightly higher initial pH (7.6) than the Kawasaki mud (pH 7.3). However, unlike the Kawasaki mud, the suspension pH of Yurakucho silt sharply drops from about 6.6 to almost 3.0 when acid input exceeds 30 cmol H_2SO_4 kg^{-1} soil. In contrast, the titration curve of the Kawasaki mud shows that the soil suspension can resist acid input with small changes in pH - dropping slowly from a pH value of 7.3 to a value of 6.0. It is only when the amount of acid reaches and exceeds as much as 130 cmol H_2SO_4 kg^{-1} soil that the suspension pH begins to decrease with a slightly faster rate, reaching its minimum value of 4.3 at the end of the test.

Taken as a whole, the titration curves show that the Kawasaki mud has a significantly higher resistance to pH changes than the Yurakucho silt. This finding can be attributed to the differences in the clay mineral composition of the studied soils. The Kawasaki mud contains montmorillonite, a mineral that has a higher cation exchange capacity (C.E.C.) value (Mitchell, 1993) than kaolinite in the Yurakucho silt. As the adsorption of H^+ ions in the studied soils is primarily through the cation exchange process, montmorillonite will be able to absorb a greater amount of H^+ before any significant changes occur in the pH values (Yong et al., 1990). In addition, the organic matter that might be present in the Kawasaki mud could also increase the buffer capacity of this soil.

4.2. Triaxial compression tests

The results of undrained triaxial tests are shown in Figs. 4 and 5, and discussed below. The effective stress paths for all tests are presented on the p'−q diagram in Figs. 4(a), 5(a), and 5(b), where $p'=(\sigma_1'+2\cdot\sigma_3')/3$, effective mean normal stress, is assigned to the horizontal axis, and $q=(\sigma_1-\sigma_3)$, deviator stress, is plotted on the vertical axis. Results of tests on soil samples consolidated to a confining pressure (σ_3') of 100 kPa are presented in the forms of deviator stress, q, against axial strain for Kawasaki mud (Fig. 4b) and Yurakucho silt (Fig. 5c). The details of the tests are summarized in Table 2, where e_i denotes the void ratio of specimens at the beginning of shearing.

The results of undrained triaxial tests on nonleached specimens of Kawasaki mud (saturated with distilled water (DW) at pH=7.3) were used as a reference. The effective stress paths obtained for different confining pressures indicate similar behavior; that is, the specimen initially contracts, generating positive pore-water pressure (the effective stress path leans throughout the loading to the left of vertical), passes through a phase-transformation state, and then dilates with the same stress ratio. The failure points from these three tests lie on a line (the critical state line) that passes through the origin of the p′-q chart. The slope of this critical state line M is about 1.58, which corresponds to a friction angle of 38.6°.

It is evident from Fig. 4a that the behavior of leached specimens is different from that of nonleached. Comparisons made between the results from these two series of tests indicate that: 1) the pore-water pressure buildup in the leached specimens (pH=6.0) is less than that of the corresponding specimens with distilled water (pH=7.3); and 2) the deviator stress (q) at failure is higher than that of the corresponding nonleached specimens (Fig. 4b).

Data obtained from a series of experiments on Yurakucho silt clearly show the effect of acidic contamination on the strength characteristics of soil, as well. Focusing first on the results of tests on nonleached specimens (saturated with distilled water, pH=7.6), it is seen that the behavior of soil is similar to that of the nonleached specimens of Kawasaki mud. A critical-state line for these tests can be obtained with a slope M=1.5; the corresponding critical-state friction angle is 36.8°. Similar to the Kawasaki mud, a slight decrease in pH from 7.6 to 6.0 would also produce specimens with the deviator stress (q) at failure higher than the corresponding nonleached samples (Fig. 5c). However, further decrease in pH significantly alters the strength characteristics of soil. Compared to the behavior observed for nonleached samples, the specimens with pH=4.0 exhibit more contractive tendencies, and yield lower values of q at failure (Fig. 5c).

4.3. Discussion

The available literature suggests that the properties of soil with different pore fluids can be governed by mechanical factors (such as physical forces acting on soil particles (Meegoda and Ratnaweera, 1994)) as well as physicochemical factors (such as changes in environmental conditions (Sridharan and Prakash, 1999)). The effect of pH on the geotechnical characteristics of clay has been reported by D'Appolonia (1980), Kashir and Yanful (2001), Wang and Siu (2006b), Gajo and Maines (2007), and Gratchev and Towhata (2011) who noted that changes in soil behavior caused by pH strongly depend on the mineral composition of the clay fraction. For soils with kaolinite, changes in pH environment will mostly affect the charge on the edges of clay particles while for soils containing smectite/montmorillonite, the influence of pH will primarily be seen in changes of the diffuse double layer (Van Olphen 1991; Mitchell 1993).

It is interesting to note that despite the difference in the clay mineralogy of the studied soils, some increase in the strength was observed for both Kawasaki mud and Yurakucho silt when pH of the pore water decreased to 6.0. Considering the available literature reviewed in section 2, it can be hypothesized that an increase in the concentration of H^+ in the pore fluid (as pH decreases from neutral to slightly acidic) would depress the thickness of the diffuse double layer, thus reducing the repulsion force between clay particles. As a result, the net of attractive van der Waals forces will increase (Anandarajah and Zhao 2000), leading to the formation of larger clay aggregates, in which clay particles are placed closer to each other. Such arrangements are expected to produce "denser" clay microfabrics with a lower initial void ratio (Table 2), which can impart additional strength to the overall soil structure. Gajo and Maines (2007) provided more support to this assumption when the investigators experimentally demonstrated that the residual shear strength of bentonite increased after it was placed in acidic environment.

Results of the tests on the Yurakucho silt given in Figs. 5(b,c) revealed that a further decrease in pH can, in fact, have the opposite effect on the strength characteristics of soil. As the pH of the pore fluid became 4.0, the undrained strength of Yurakucho silt decreased compared to the one that was obtained for the nonleached specimens (pH=7.6). Such changes in the soil behavior can be attributed to the further transformation of the clay microfabric. In particular, when pH<5.5, the charge on the edges of kaolinite particles becomes increasingly positive, a process that leads to the formation of more open, flocculated structures (Van Olphen 1991, Mitchell 1993). Sridharan et al. (1988) noted that such flocculated arrangements of kaolinite particles would enclose large spaces for water entrapment and thus exhibit higher liquid limit values. This study provides some support for this assumption as the obtained data from Atterberg limits tests (Table 1) show that the liquid limit of Yurakucho silt indeed increased after the soil was leached with acidic fluids to a low pH of 4.0. Furthermore, Wang and Siu (2006b) pointed out that flocculated clay arrangements of kaolinite particles at low pH are associated with overall “looser” soil structures and lower strength of soil.

Considering the aforementioned discussion, it can be hypothesized that, at pH=4.0, the leached specimens of Yurakucho silt formed more open, flocculated structures (with a greater initial void ratio (Table 2)) that had the tendency to contract more and generate higher pore-water pressures when subjected to loading.

5. Conclusions

Two soils with different clay mineral composition were leached with acidic fluids for a significant period of time. The properties and strength characteristic of such soils were studied and the following conclusions were made:

- The buffer capacity of Kawasaki mud was found to be higher than that of Yurakucho silt due to the presence of montmorillonite and, possibly, organic matter.
- The strength of Kawasaki mud and Yurakucho silt slightly increased when the pH of pore fluids decreased to 6.0.
- The strength of Yurakucho silt significantly decreased at low pH conditions (pH=4.0) which resulted in smaller values of deviator stress at failure compared to the nonleached samples.

This study indicates that the effect of acidic fluids on the strength of soil is complex, and further research is needed to clarify the findings presented in this paper.

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Figure captions:

Figure 1. A sketch of a container used to leach soil with acidic fluids.

Figure 2. Changes in the liquid's pH over almost a 300-day period for the Yurakucho silt and Kawasaki mud.

Figure 3. pH-titration curves of soils studied.

Figure 4. Undrained behavior of Kawasaki mud with distilled water and leached with acidic fluids. Effective stress path (a), and stress-strain relationship (b). (σ_3' - effective confining stress).

Figure 5. Undrained behavior of Yurakucho silt with distilled water and leached with acidic fluids. Effective stress path for specimens saturated with water (a), effective stress path for specimens leached with acidic fluids (b), and stress-strain relationship (c). (σ_3' - effective confining stress).