A PHYSICO-CHEMICAL APPROACH TO THE
CONSOLIDATION MECHANISM OF SOFT CLAYS

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ABSTRACT

An experimental attempt was made to distinguish between primary and secondary phases of consolidation of soft clays under incremental and continuous loading. The consolidation mechanism of soft clays under elevated temperature conditions was also investigated.

Making use of the differences in the replaceability of exchangeable cations absorbed to the clay particles, a special method of preparation of the clay samples was developed. The time-dependent variations of concentrations of the particular types of cations Ca^{2+} and Na^+ which existed in the pore fluid drained from clay samples during consolidation were obtained from quantitative chemical analyses.

The greater increases in the concentrations of divalent cations Ca^{2+} contained in the pore fluid within the micro-pores were observed during both secondary and elevated temperature consolidation. These increases were considered to demonstrate the progressive deformation of micro-pores within the clay microstructures. The volume change of clay under constant rate of strain loading was presumably due to the deformation of the macro-pores. The main cause of "aging effects" from elevated temperature consolidation was shown to be the acceleration of secondary compression of the clay, which would have occurred during long term consolidation under normal temperature conditions.

Key words: clay, consolidation, physico-chemical properties, soil structure, soil water, temperature effect (IGC: D5/D2/D3)

INTRODUCTION

Recently, the possibility of duplicating the natural aging of marine clays by consolidating clay slurry at high temperature and cooling it after the completion of consolidation was proposed. The main cause of the effects of high temperature consolidation is assumed to be the acceleration of cementation (cementing action) between the mineral particles (Tsuchida et al., 1991).

Consolidation phenomena of saturated clays are defined by the time-dependent volume decrease due to the drainage of pore water existing in clays and conveniently divided into two phases, as shown in Fig. 1. From the micro-structural point of view, it is generally considered that the primary phase of consolidation of clay is due to the drainage of pore fluid existing in the macro-pores among the peds (aggregates of clay particles). The secondary phase of consolidation of clay is considered to be mainly due to the delayed deformation of micro-pores within the peds according to the viscous flow of pore fluid closely adsorbed to the surfaces of clay particles (Matsuo and Kamon, 1977).

Thus it is presumed that the pore fluid or particular types of cations existing in the micro-pores at the beginning of consolidation are drained during the secondary consolidation of soft clay. If the clay samples are initially prepared to contain the particular types of cations within the pore fluid of micro-pores, these particular types of ca-
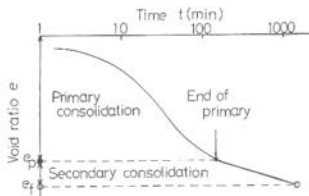

Fig. 1. Primary and secondary phases of consolidation of soft clay

tions can be assumed to be used as an indicator of the progressive deformation of micro-pores during the secondary phase of consolidation. It becomes possible therefore to distinguish between the primary and secondary phases of consolidation, if the quantitative chemical analyses on the pore fluid drained during consolidation of specially prepared clay samples, are carried out.

In this paper, a discussion of the experimental attempt to distinguish between the primary and secondary phases of consolidation of soft clays under incremental and continuous loading conditions by means of the quantitative chemical analyses on the pore fluid drained from specially prepared clay samples is made. Based on the experimental results favorable to the possibility of determining the secondary phase of consolidation, the cause of high temperature consolidation effects assumed by Tsuchida et al. (1991) was also investigated with regard to the consolidation mechanism of soft clay under high temperature conditions.

REVIEW OF PREVIOUS PHYSICO-CHEMICAL APPROACHES

Exchangeable Cations and Their Replaceability

According to Mitchell (1993), a clay adsorbs cations with a fixed total charge under a given set of environmental conditions (temperature, pressure, pH, total electrolyte concentration). The most commonly found cations in soils are calcium (Ca^{2+}), magnesium (Mg^{2+}), sodium (Na^+) and potassium (K^+). These cations can be replaced with a group of different ions having the same total charge, by means of isormorphous substitution, broken bonds and replacement (Grim, 1968).

Ions of one type can be replaced by ions of another type; for example, Ca^{2+} for Na^+, Na^+ for Ca^{2+}, Fe^{3+} for Mg^{2+}, and so on. The ease with which an ion of one type can replace an ion of another type depends mainly on the valence, relative abundance of the different ion types and ion size. A typical replaceability series is as follows.

$$Na^+ < Li^+ < K^+ < Rb^+ < Cs^+ < Mg^{2+} < Ca^{2+}$$
$$< Ba^{2+} < Cu^{2+} < Al^{3+} < Fe^{3+} < Th^{4+}$$
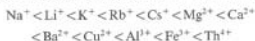
A number of theories have been proposed for the quantitative description of the equilibrium concentrations of different cations in the adsorbed layer in terms of concentrations in the bulk solution. For practical purposes the *Gapon equation* is introduced to be useful for assessing the proportions of monovalent and divalent ions, except in highly acid soils. If the subscript s refers to the exchange complex of the soil, the subscript e refers to the equilibrium solution, M and N are monovalent ion concentrations and P refers to the concentration of divalent ions, then we have

$$\left(\frac{M^+}{N^+}\right)_s = k_1 \left(\frac{M^+}{N^+}\right)_e$$

and

$$\left(\frac{M^+}{P^{2+}}\right)_s = k_2 \left[\frac{M^+}{(P^{2+})^{1/2}}\right]_e$$

where k_1 and k_2 are selectivity constants.

As an example, if the proportions of Na^+ and Ca^{2+} in the adsorbed complex are equal and the concentration of Na^+ in the free solution is doubled, then the concentration of Ca^{2+} in the free solution must be quadrupled if the proportions of adsorbed ions are to remain the same.

If the composition of the pore fluid is known, the relative amounts of monovalent and divalent ions in the adsorbed cation complex can be estimated by these equations.

Microstructures of Clays

According to the observations of the soil micro-structure using the scanning electron microscope, it has been recognized that both artificial and natural clay soils are made up of clay particles aggregated into "peds" (Collins and McGown, 1974; Matsuo and Kamon, 1977; and so on). It is also assumed that these aggregated clay particles are the minimum units controlling the macroscopic mechanical behavior of clay soils. The terms "peds and pores" for the basic units constituting the microstructure have been defined by Matsuo and Kamon (1977), as shown in Table 1.

Effects of Temperature on Consolidation of Clays

Temperature variations can cause significant changes in the volume and effective stress of saturated soils. Mitchell (1993) describes the detailed investigations on the effects of temperature variations on the volume change behavior of a saturated soil water system. It is concluded therefore that partial collapse of the soil structure and a decrease in void ratio occurs until a sufficient number of additional bonds are formed to enable the soil to carry the stress at the higher temperature. This effect is assumed to be analogous to secondary compression under a stress increase. Similar effects of elevated temperature on the volume change behavior of fine grained soil and physico-chemical approaches to these temperature effects are described by Passwell (1967) and Plum and Esrig (1969).

According to Tsuchida et al. (1991), remolded clay samples, whose mechanical properties are similar to that of lightly aged clay, can be produced easily in the laboratory by consolidating clay slurry at a high temperature (75°C) and cooling it after the completion of consolida-

Table 1. Peds and pores defined (Matsuo and Kamon, 1977)

macroped	(>50 μm) macropore (>10 μm)	by naked eye and spy glass
mesoped	(2~50 μm) mesopore (1~10 μm)	by optical microscope
microped	(0.1~2 μm) micropore (0.01~1 μm)	by scanning electron microscope
submicroped	(<0.1 μm) submicropore (<100 Å)	by transmission electron microscope

tion. They concluded that the acceleration of the cementation was the main cause of the effect of the high temperature consolidation.

CLAY SAMPLES AND A SPECIAL METHOD OF PREPARATION

Commercial bentonite clay materials were used in this study. The physico-chemical properties of these materials are listed in Table 2.

In order to prepare the clay samples which contain different types of cations in the pore fluid in macro-pores and micro-pores, a special method of preparation was adopted for this study, making use of the differences in the replaceability of exchangeable cations. The method of preparation for the clay samples was as follows.

Bentonite clay materials, 50 (g), were thoroughly mixed with 250 (ml) of calcium chloride ($CaCl_2$) whose concentration was 1 (mol/l) and maintained at saturation for 1.5 (hours). The calcium ions Ca^{2+} replaced other types of cations which pre-existed on the surfaces of the clay particles, so that the major types of cations adsorbed to clay particles became Ca^{2+}. A paste of the clay samples was mixed and remolded repeatedly with ethyl alcohol over boiling water to remove the excessive Ca^{2+}. Finally the clay pastes were mixed with 100 (ml) of sodium chloride NaCl whose concentration was 0.01 (mol/l). Through these procedures clay samples were prepared, whose macropores contained the pore fluid with Na^+ and micro-pores contained the pore fluid with mainly Ca^{2+}. The range of magnitudes (meq/100 g) of two types of cations Ca^{2+} and Na^+ adsorbed to the clay samples prepared by this special method of preparation are also listed in Table 2, dependent upon the number of repetitions of the removal of excessive Ca^{2+}.

CONSOLIDATION TESTS AND CHEMICAL ANALYSES OF DRAINED PORE FLUID

The consolidometer used in this study is shown in Fig. 2. The internal diameter of the consolidation ring is 100 (mm) and the drainage line is directly connected to the base plate, so that the pore fluid drained during consolidation could be extracted directly from the drainage line as quickly as possible. The consolidometer and other devices were made of acrylic resin to avoid the possibility of rusting of metals and including the cations dissolved from the consolidation metal ring in the drained pore

Table 2. Physico-chemical properties of clay sample

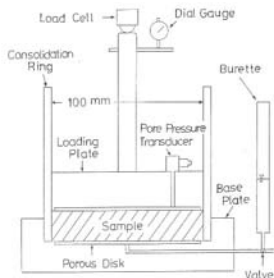|	
Specific gravity G_s	2.90
Liquid limit w_L (%)	152.2
Plastic limit w_P (%)	46.3
Plasticity index I_P	105.9
Cation exchange capacity (meq/100 g)	122.70
Ranges of adsorbed cation concentrations	
Ca^{2+} (meq/100 g)	81.64 – 108.63
Na^+ (meq/100 g)	7.90 – 37.23

Fig. 2. Consolidometer used in this study

fluid. In the case of high temperature consolidation, this consolidometer was submerged into a hot water bath, whose temperature was kept almost constant at 70(°C).

The consolidation pressure was increased from 9.8 (kPa) in steps up to the magnitude of 78.5 (kPa) in the case of incremental loading. The time for consolidation under one increment of loading pressure was 24 (hours) and the single drainage condition was provided at the bottom of the specimen. Quantitative chemical analyses on the drained pore fluid sampled a few times during the consolidation process were carried out by using inductively coupled plasma spectroscopy (ICP), whose accuracy for measuring the concentrations is ±5(%) of the measured value.

Incremental Loading under Room Temperature

The time variations of void ratio e of the clay sample and the changes in the concentrations of two types of cations Na^+ and Ca^{2+} contained in the drained pore fluid are shown in Fig. 3, for the test with the consolidation pressure p=39.2 (kPa). The test results under a consolidation pressure of p=78.5 (kPa) are also shown in Fig. 4. The numbers in these figures indicate the sampling sequences, when the pore fluid drained from clay sample was sampled and analyzed chemically.

The values of the concentrations of both types of cations gradually increased during the process of consolidation. In these figures the points of completion of primary consolidation t_E determined by the method of 3 times to t_T are indicated (Oda and Mitachi, 1988). The elapsed time at sample number 6 is much greater than that of t_E, so that the phase of consolidation at this point is considered to be the secondary phase. The change of Ca^{2+} concentration becomes greater than that of Na^+ particularly in this secondary phase. Since Ca^{2+} is mainly contained in the pore fluid of micro-pores, the greater increase in Ca^{2+} concentration is considered to demonstrate the progressive deformation of micro-pores of soft clays dur-
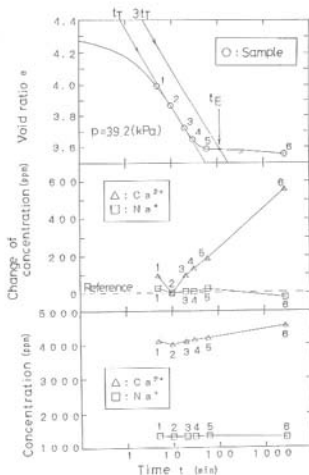
Fig. 3. Relationships for void ratio e and the changes of Ca^{2+} and Na^+ concentrations under incremental loading ($p=39.2$ (kPa))
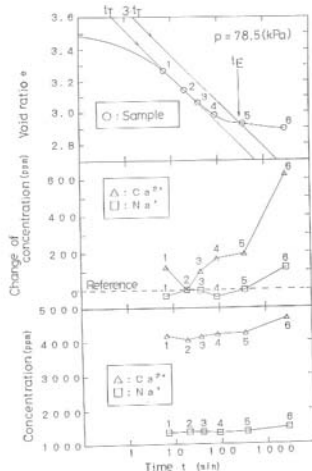

Fig. 4. Relationships for void ratio e and the changes of Ca^{2+} and Na^+ concentrations under incremental loading ($p=78.5$ (kPa))

ing the secondary phase of consolidation. It is assumed that the viscous flow of pore fluid adsorbed to the surfaces of clay particles due to the deformation of micropores could occur during the primary phase of consolidation, because the concentration of Ca^{2+} increased gradually during this primary phase. In these figures, the changes of cation concentrations are obtained by making reference to the values of sample number 2, since the values of sample number 1 are considered to be influenced by the pore fluid during secondary consolidation at the previous consolidation pressure.

In this case however it is necessary to take into account the equilibrium of the exchangeable cation concentrations obtained by the *Gapon equation* described before. According to *Gapon equation*, the increase of the concentration of Ca^{2+} existing in the drained pore water must inevitably be followed by an increase in the concentration of Na^+. If the Ca^{2+} concentration existing in the drained pore fluid is quadrupled, then the Na^+ concentration must be doubled in order to maintain the equilibrium between the adsorbed exchangeable cation concentrations. Although the concentration of Na^+ existed in the expelled pore fluid increased during consolidation, the magnitude of the increase in concentration of Ca^{2+} is much greater than the value predicted by the *Gapon equation*.

Continuous Loading under Room Temperature

Constant rate of strain (CRS) consolidation tests were carried out to investigate the consolidation behavior of soft clay under continuous loading condition. The CRS tests were performed using the same consolidometer as in Fig. 2, equipped with the measurement system for pore water pressure developed at the top impervious end of the specimen. After the incremental consolidation up to a consolidation pressure of $p=39.2$ (kPa), the axial loading pressure was continuously increased to 78.5 (kPa) under the controlled strain rate of the specimen of 0.07(%/min). After the completion of CRS loading, the consolidation of soil was allowed under the approximately constant axial loading pressure of 78.5 (kPa) for 2 (days). The Axial stress-pore pressure-axial displacement changes of soft clay during CRS consolidation test are shown in Fig. 5. The numbers in this figure also indicate the sequences of sampling for the drained pore fluid for chemical analyses. After the completion of CRS loading, the excess pore pressure quickly dissipated and the consolidation progressed under almost constant consolidation pressure, when the axial loading pressure was kept constant. During this process, the magnitude of excess pore water pressure fluctuated a little due to the change in room temperature.
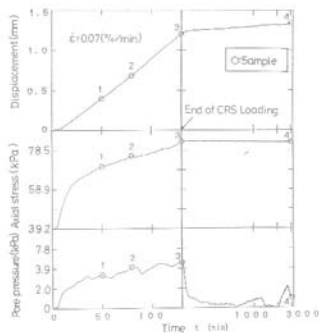
Fig. 5. Axial loading stress–pore pressure–axial displacement behavior under continuous loading ($\varepsilon = 0.07(\%/\text{min})$)

Fig. 6. Relationships for void ratio e and the changes of Ca^{2+} and Na^+ concentrations under continuous loading

The changes of the void ratio e of clay sample and of the concentrations of two types of cations Na^+ and Ca^{2+} with the consolidation pressure p are shown in Fig. 6, under the CRS loading and constant axial stress condition. In this figure, the changes of cation concentrations are obtained by making reference to the values at the end of primary consolidation under previous consolidation pressure.

During CRS loading, the magnitudes of the concentration changes of two types of cations were very small. However the value of Ca^{2+} concentration suddenly increased during constant axial stress, in contrast to the almost constant value of Na^+ concentration. It is considered that no viscous flow of pore fluid in micro-pores could occur during CRS loading, thereafter the pore fluid in micro-pores would flow viscously under the constant consolidation pressure.

Primary and Secondary Consolidation under Room Temperature

Based on these experimental results, the values of cation concentration increases ΔCa^{2+} and ΔNa^+ versus the void ratio decreases Δe during primary and secondary consolidation can be obtained as follows. Since the change of the values of ΔCa^{2+} is considered to demonstrate the deformation of micro-pores of soft clays during secondary phase of consolidation, the quantitative evaluation of the microstructural change of soft clays would be possible by observing the change of ΔCa^{2+}.

For example, it can be seen from Fig. 3 that the value of ΔCa^{2+} obtained between samples numbers 2 and 5 during primary consolidation is 200 (ppm) and the value of Δe during the same period is 0.3. Accordingly, the value of $\Delta Ca^{2+}/\Delta e$ is equal to $200/0.3 \doteq 6.7 \times 10^2$.
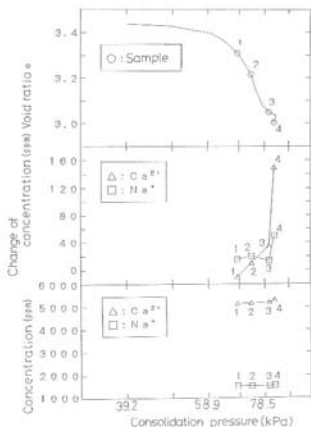
The value of $\Delta Ca^{2+}/\Delta e$ between samples numbers 5 and 6 corresponding to secondary consolidation is $400/0.02 \doteq 2.0 \times 10^4$. Hence the value of $\Delta Ca^{2+}/\Delta e$ during secondary consolidation becomes about 30 times the value during primary consolidation. A similar relationship is obtained from Fig. 4.

In the case of continuous loading, the value of $\Delta Ca^{2+}/\Delta e$ between samples numbers 1 and 3 during primary consolidation obtained from Fig. 6 is $40/0.25 \doteq 1.6 \times 10^2$ and the value of $\Delta Ca^{2+}/\Delta e$ between 3 and 4 corresponding to secondary consolidation is $120/0.03 \doteq 4.0 \times 10^3$. Hence the value of $\Delta Ca^{2+}/\Delta e$ during secondary consolidation becomes about 25 times the primary consolidation value and the difference of $\Delta Ca^{2+}/\Delta e$ between secondary versus primary consolidation is almost the same as for the incremental loading.

It is concluded therefore that the secondary phase of consolidation of soft clay could occur due to the deformation of micro-pores and the volume change of clay under constant rate of strain loading is mainly due to the deformation of macro-pores between macro-peds. Since the accuracy for measuring the cation concentrations is within ±5(%) of the measured value, the discussions mentioned above are limited to the cases where the measured values of the change of ΔCa^{2+} are greater than the value of ±5(%) of the measured.
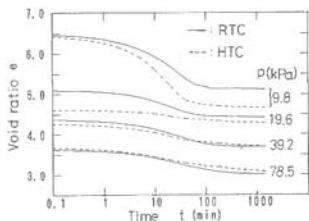
Fig. 7. Incremental compression curves under room and high temperature conditions
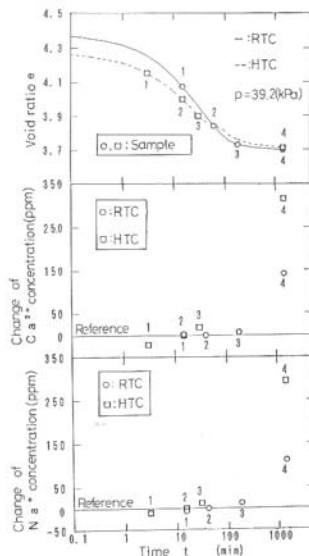

Fig. 8. Relationships for void ratio e and the changes of Ca^{2+} and Na^+ concentrations under room and high temperature conditions ($p=39.2$ (kPa))
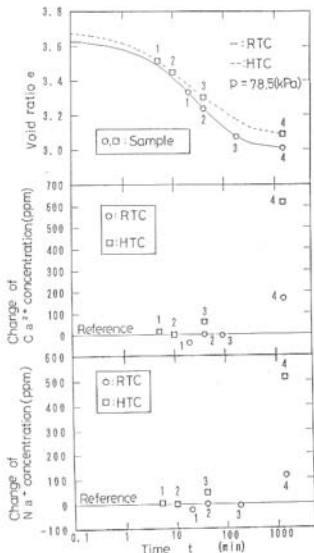

Fig. 9. Relationships for void ratio e and the changes of Ca^{2+} and Na^+ concentrations under room and high temperature conditions ($p=78.5$ (kPa))

Incremental Loading under Elevated Temperature

Figure 7 compares the void ratio e of clay sample versus elapsed time t curves under room temperature (RTC) and high temperature (HTC). It was obvious that these compression curves were different during the initial stages of loading, however the differences in void ratio became small as the consolidation pressure p increased. As a result, the final void ratio of the room temperature sample was 3.01 and the final value of high temperature sample was 3.09.

Figure 8 shows the relationships among void ratio e of clay sample and the changes of the concentrations of two types of cations Na^+ and Ca^{2+} contained in the drained pore fluid versus the elapsed time t under the room temperature and the high temperature at consolidation pressure of $p=39.2$ (kPa). Similar relationships for the different temperature conditions at consolidation pressure of $p=78.5$ (kPa) are shown in Fig. 9.

There are only little differences in the decreases of the

void ratio during secondary phases of consolidation between the room temperature and high temperature consolidation. The increases in the concentrations of two types of cations contained in the drained pore fluid under high temperature condition however became much greater than those of the concentrations under room temperature condition.

Based on these Figs. 8 and 9, it is possible to carry out quantitative evaluation of the consolidation mechanism of soft clay under elevated temperature condition by using the similar values of $\Delta Ca^{2+}/\Delta e$ during primary and secondary phases of consolidation as described for Figs. 3, 4 and 6. In the case of RTC consolidation, the value of $\Delta Ca^{2+}/\Delta e$ between samples numbers 2 and 3 corresponding to primary consolidation obtained from Fig. 8 is $10/0.1 = 1.0 \times 10^2$ and the value of $\Delta Ca^{2+}/\Delta e$ between 3 and 4 corresponding to secondary consolidation is $130/0.04 \doteq 3.3 \times 10^3$. Hence the value of $\Delta Ca^{2+}/\Delta e$ during secondary consolidation becomes about 30 times the value during primary consolidation and the difference of $\Delta Ca^{2+}/\Delta e$ between secondary versus primary consolidation is almost the same as described in the previous section.

In the case of HTC consolidation, however the value of $\Delta Ca^{2+}/\Delta e$ during primary consolidation obtained from Fig. 9 is $60/0.15 = 4.0 \times 10^2$ and the value of $\Delta Ca^{2+}/\Delta e$ during secondary consolidation is $560/0.2 = 2.8 \times 10^3$. The value of $\Delta Ca^{2+}/\Delta e$ during primary consolidation in the case of HTC consolidation is 4 times the value of $\Delta Ca^{2+}/\Delta e$ in the case of RTC consolidation. In addition the value of $\Delta Ca^{2+}/\Delta e$ during secondary consolidation in the case of HTC tests is about 7 times the values of $\Delta Ca^{2+}/\Delta e$ during primary consolidation. Consequently, the secondary consolidation of soft clay is considered to be accelerated by the elevation of ambient temperature.
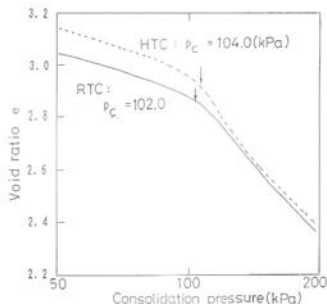

Fig. 10. Comparison between $e - \log p$ curves for room temperature and high temperature samples

After the completion of incremental consolidation tests, the CRS consolidation tests of 0.01(%/min) strain rate were carried out for the room temperature consolidation sample and the high temperature consolidation sample. The relationships between void ratio e and consolidation pressure $\log p$ of room temperature sample and high temperature sample obtained from these CRS consolidation tests are compared in Fig. 10. The initial values of void ratio for two types of samples after incremental loading and unloading are a little different. The consolidation yield stress p_c of the high temperature sample is however a little greater than that of the room temperature sample and its compression index C_c defined by the gradient of $e - \log p$ curve decreases rapidly near the value of p_c. These aging effects are almost the same as reported by Tsuchida et al. (1991). They assumed that the main cause of such "aging effects" of high temperature consolidation was the acceleration of cementation (cementing action) between the clay mineral particles.

The clay particles are bonded together by the cementing agents, i.e. calcium carbonates $CaCO_3$ and magnesium carbonates $MgCO_3$. If the main cause of "aging effects" of high temperature consolidation should be the accelerated cementation, the concentration of Ca^{2+} drained during high temperature consolidation should decrease to a smaller value than that of room temperature consolidation because of the production of calcium carbonates $CaCO_3$. It is difficult therefore to assume the main cause of "aging effects" of high temperature consolidation as the accelerated cementing action, since the concentration of Ca^{2+} within the pore fluid drained during high temperature consolidation were larger than those for the room temperature consolidation.

Adsorbed water molecules on the clay particles are bonded by the hydrogen bonds between the oxygen atoms existing on the clay mineral surfaces and the water molecules. These hydrogen bonds can easily be broken under the elevation of temperature up to 70($°C$). The exchangeable cations Ca^{2+} existing in these broken adsorbed water layers may displace so easily that the concentration of Ca^{2+} in the drained pore fluid increases more rapidly than that for room temperature consolidation. After the completion of high temperature consolidation of the clay and cooling, the microstructure of the clay becomes rigid enough due to mineral-mineral contacts between particles at room temperature, as if it were subjected to natural long term secondary compression. It is possible therefore to assume that the main cause of "aging effects" of elevated temperature consolidation is the acceleration of secondary compression of clay, which would have occurred during long term consolidation at normal temperature condition. Similar arguments on the mechanism of secondary compression under the elevated temperature have been described by Towhata et al. (1993).

CONCLUSIONS

An experimental attempt was made to distinguish be-

tween the primary and secondary phases of consolidation of soft clays under incremental and continuous loading conditions by using the quantitative chemical analyses on the pore fluid drained from specially prepared clay samples. Based upon the experimental results obtained in this study, the cause of the "aging effects" of high temperature consolidation was also investigated.

It is concluded from the experimental results that:

(1) It is possible to prepare the clay samples which contain the Ca^{2+} within the micro-pores of clay and the Na^+ within the macro-pores by making use of the differences in the replaceability of the two types of cations. Consequently these two types of cations can be used as an indicator of the progressive deformation of micro-pores during the secondary phase of consolidation.

(2) It is possible to distinguish between the primary and secondary phases of consolidation of soft clays under incremental and continuous loading conditions by using the quantitative chemical analyses on the pore fluid drained from the specially prepared clay samples described above.

(3) Secondary phase of consolidation could occur due to the deformation of micro-pores and initiate from the primary phase of consolidation.

(4) During the constant rate of strain (CRS) loading, the compression of soft clay is mainly due to the deformation of macro-pores between macro-peds.

(5) The main cause of "aging effects" from elevated temperature consolidation is the acceleration of secondary consolidation of clay, which would have occurred during long term consolidation at normal temperature conditions.

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