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# Investigation on Microstructure and Strength of Lime and Cement Stabilized Ariake Clay

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**ABSTRACT :** Lime and cement are effective admixtures that are presently applied to stabilize soft clay. To investigate the microstructure and strength of lime and cement stabilized clays, an experimental study was conducted. Atterberg limit, scanning electron microscope (SEM), permeability, unconfined compression and oedometer tests were performed on Ariake clays mixed with lime and cement. The results show that the strength development relates to the microstructure, which is fabric and cementation. The lime and cement stabilized non-sulfide clays clearly show the presence of the cementing products. Strengths of both stabilized-clays increase with curing time and admixture contents. The lime-stabilized clay, which contains high sulfide content, the cementing products are invisible and the strength is extremely low.

# **INTRODUCTION**

Disposal of surplus soils at construction sites has become an urgent problem as it is becoming increasingly difficult to secure dumping sites. Therefore, it is necessary to recycle the surplus soils by relatively simple treatment such as chemical stabilization. Lime and cement are the effective admixtures for stabilization. Adding lime or cement to soft clay improves its engineering properties such as index properties, strength and resistance to compressibility. The successful application of lime and lime mixed with waste materials such as fly ash, ark shell ash and foamed waste glass stabilized soft Ariake clay were reported by Nanri and Onitsuka (1996) and Onitsuka and Shen(1998, 2000). The lime and cement stabilized surplus clays are normally utilized as engineering materials for backfill and pavement. The strength development of lime and cement stabilized clay are mainly obtained by formation of cementing products (CaO-SiO<sub>2</sub>-H<sub>2</sub>O, CaO-Al<sub>2</sub>O<sub>3</sub>-H<sub>2</sub>O) that were investigated by x-ray diffraction and scanning electron microscope (SEM) (Kawamura and Dimond 1975; Kamon and Nontananandh 1991 and Rajasekaran et al.1997). It is clearly shown that the cementing products cause the strength of stabilized clay to increases. There are many factors such as differences in soil gradation, types of clay minerals, organic matter, pH, sulphate and etc., (Sherwood 1958, 1962; Moh 1962; Mateos 1964; Thompson 1966 and Miura et al.1988) that significantly in-

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fluence the ability of clay to react with lime and cement to achieve a strength increase.

Ariake clay is a kind of very high sensitive clay that deposited around the coast of the Ariake Bay in Kyushu Island (Nakamura et al.1985). The clay near river sites contains high sulfide content because the sulfate ion occurs naturally in most water supplies and is present in wastewater as well. Sulfur is required in the synthesis of proteins and is released in their degradation. Sulfate is reduced biologically under anaerobic conditions to sulfide. The sulfur cycle in soft clay was also investigated by Michell (1984).

The study investigates on microstructure, strength and compressibility of lime and cement stabilized clays. The microstructure feature of the stabilized soft clays was observed by an Atterberg limit, scanning electron microscope (SEM) and permeability tests. The tests, carried out to study the strength development and compressibility of stabilized soft clays were the unconfined compression and oedometer tests, respectively.

# EXPERIMENTAL INVESTIGATION

Ariake clays used in this investigation were obtained form Okawa area, Fukuoka prefecture and Ashikari area, Saga prefecture designated as Clay 1 and Clay 2, respectively. Clay 1 was sampled at 1.0 m depth under the bottom of the rivulet. Clay 2 was sampled at 3.0 m depth from the ground surface. Properties of clay samples were shown in Table 1.

Sample	Clay 1 Okawa Area	Clay 2 Ashikari Area
Natural water content, %	185.0	150.0
Liquid limit, %	142.7	133.0
Plasticity index	89.1	71.4
Specific gravity	2.48	2.62
Particle size distribution, %		
(2.000 - 0.075 mm) Sand	3	1
(0.075 - 0.005 mm) Silt	52	44
(<0.005 mm) Clay	45	55
Salt concentration (g/l)	0.0	8.0
pH	6.0	7.6
Organic content, %	1.0	1.0

Table 1. Properties of Ariake clays

To identify clay type, the free swell technique that is sediment volumes of 10 g dry soil in 100 ml of water ( $H_2O$ ) and carbon tetrachloride (CCl<sub>4</sub>) was used (Sridharan et al.1986).

To investigate the effect of lime addition on consistency limits, the clays were mixed with 5, 10, 15, 20 and 30 % lime and Atterberg limit tests were conducted immediately after being mixed. The tests were also conducted on 10% lime stabilized clays at the curing periods of 7,14 and 28 days.

# Microstructure

The micrograph is one of the effective observations on the microstructure of the stabilized clay. Clay specimens were prepared by freeze drying method for minimizing the volume change (Onitsuka et al.1998, 2001) and the fractured surface of the specimens were coated with gold to view the soil structure and the nature of the products formed. The microstructures of samples obtained are discussed in the following sections.

The coefficient of permeability is one of the mechanical parameters for analysis of the structural properties of soil. The falling head permeability test on remolded clays and stabilized clays at curing time 7 days were carried out at the end of each loading of the oedometer test. The oedometer apparatus was connected to a burette by a tube, and water flowed from the burette to the bottom of the sample.

# Strength and compressibility

Quick lime and ordinary portland cement were used for the stabilization. The water content of the all clay samples which were mixed with 5, 10, 20% lime and 10, 20, 30% cement by weight of dry soil are 185%. Distilled water was added in Clay 2 to adjust the water content to 185%. All samples were compacted by hand vibrating to eliminate the entrapped air. The specimens, cured at a temperature about 20°C and a humidity 90%, were 50 mm in diameter and 100 mm in height. The unconfined compression tests were conducted at the curing periods of 7, 14 and 28 days.

The series of oedometer tests were carried out on the remolded and stabilized clays at the curing time 7 days. The specimens were the remolded clays and stabilized clays, which were mixed with lime and cement at the same initial water content as 185%.

## **Retarding factors**

The tests were performed to investigate the effect of clay mineral, organic matter and sulfide on the strength development of lime and cement stabilized clay.

To investigate organic matter effect, the inorganic Clay 1 (Clay 1 was treated by Hydrogen peroxide ( $H_2O_2$ ) and then washed several times with distilled water until the washings did not indicate acidity) was then mixed with 20% lime at the water content as 185%. The unconfined compression tests were conducted at after the curing period of 28 days.

Clay 1 was sampled from the bottom of the rivulet. It contained high sulfide content. The effect of sulfide is investgated by mixing free sulfide Clay 1 with lime and cement. To eliminate the sulfides, Clay 1 was treated by sulphuric acid (H<sub>2</sub>SO<sub>4</sub>) until no further evolution of hydrogen sulfide (H<sub>2</sub>S) occurred, and then washed several times with distilled water until the washings did not indi-

cate acidity (BS 1377 : Part 3 : 1990 : 3). The Clay 1 that was treated by chemical to eliminate sulfide is named as "Treated Clay 1". To obtain the specimens contained various sulfide contents, Treated Clay 1 was then mixed with initial Clay 1 in different proportions as 100% Treated Clay 1, 75% Treated Clay 1+25% Clay 1, 50% Treated Clay 1+50% Clay 1, 25% Treated Clay 1+75% Clay 1 and 100% Clay 1. All clay samples were mixed with 20% lime or cement at the same water content as 185% and cured 7 days. A series of oedometer tests were carried out on the remolded and stabilized clays.

## **RESULTS AND DISCUSSIONS**

The free swell technique is a rapid method to identify clay type. The plot of sediment volume soils in water versus their sediment volume in carbon tetrachloride is shown in Fig.1. The results of Clay 1 and Clay 2 show that the sediment volume in carbon tetrachloride is higher than water. This is a property typical of non-swelling clays as kaolinite and chlorite. Although Ariake clays show the high sediment volume in carbon tetrachloride, their sediment volumes in water are also higher than 2.1 cc/g that indicate the presence of the swelling smectite clay in soils. The results show that Clay 1 and Clay 2 are not so much different in clay types.



Fig.1 Sediment volume of Clay 1 and Clay 2 in water and CCl<sub>4</sub>

# Liquid limit of lime-stabilized clay

Figs.2 (a) and (b) show the liquid limit of Clay 1 and Clay 2 that were mixed with various lime contents. The liquid limit of Clay 1 decreases with the increase in lime content. The free swell test shows that the Ariake Clay 1 and Clay 2 are not so much different in clay types that are mixture of two types of clays and more influence by non-swelling clay. The liquid limits of these clays are controlled by the shearing resistance at particle level and the thickness of the diffuse double layer (Sridharan and Rao, 1975). The addition of lime in Clay 1 reduces the thickness of the diffuse double layer of clay particles by increasing the electrolyte concentration and by exchanging cation. Hence,



Fig.2 (a) Atterberg limit of Clay 1 mixed with lime at water content 185%



Fig.2 (b) Atterberg limit of Clay 2 mixed with lime at water content 185%



Fig.3 Liquid limit of clays mixed with lime at water content 185%

the addition of lime induces to decrease in liquid limit. The result shows that the thickness of the diffuse double layer is more influntial than shearing resistance. The addition lime cannot increase the attractive force to bring out the particle flocculation. Clay 1 mixed with lime cannot form cementing products (CaO-SiO<sub>2</sub>-H<sub>2</sub>O, CaO-Al<sub>2</sub>O<sub>3</sub>-H<sub>2</sub>O), which increase the shearing resistance. In the case of Clay 2, addition of 10 and 20% lime increase the liquid limit from 133 to 140 and 142, respectively. The addition of lime increases the cationic valency and then increases the interparticle attractive force, which favors higher flocculation of clay particles. The liquid limit increases due to the higher water holding capacity. That agrees well with the observation of lime stabilized clay, which shows an increase in liquid limit with lime addition (Locat et al.1996).

Fig.3 shows the relation between the liquid limit of lime stabilized Clay 1 and Clay 2 and curing time. The liquid limit of lime stabilized Clay 2 increases with curing time because of the formation of coarser aggregates and a more flocculated particle arrangement that is also reported by Locat et al.(1996). The liquid limit of lime stabilized Clay 1 is constant with curing time even though it was cured 28 days. It means that the lime stabilized Clay 1 cannot produce cementing products to increase the liquid limit.



Fig.4 (a) Remolded Clay 1 at water content 185%



Fig.4 (b) Remold Clay 2 adjusted at water content 185%



Fig.5 (a) Clay 1 mixed with 10% lime at curing time 7 days



Fig.5 (b) Clay 2 mixed with 10% lime at curing time 7 days



Fig.6 (a) Clay 1 mixed with 20% lime at curing time 7 days



Fig.6 (b) Clay 2 mixed with 20% lime at curing time 7 days

#### **Microstructure observation**

The term" fabric "refers to the arrangement of the particle, particle group, and pore spaces in a soil (Mitchell, 1993). A cluster is a grouping of particles or aggregates into larger fabric units (Olsen, 1962 ; Yong and Sheeran, 1973). Figs.4 (a) and (b) show the micrograph of remolded Clay 1, which indicates the presence of diatom, and Clay 2 at the same water content as 185%. It presents that the pore spaces in fabric of Clay 2 are bigger than Clay 1 because at the same water content, Clay 2 is in the state that liquidity index is higher than Clay 1. In Fig.5 (b), Clay 2 is stabilized by 10% lime and clearly shows the presence of cementing products (CaO-SiO<sub>2</sub>-H<sub>2</sub>O, CaO-Al<sub>2</sub>O<sub>3</sub>-H<sub>2</sub>O) in the pore system with the well-knit framework between the clusters. The formation of these cementation compounds in lime-stabilized clay was reported earlier by Kawamura and Dimond (1975). The cementing products bond each cluster together, like bridge between clusters. It is called "bound bridge". Fig.5 (a) shows the micrograph of lime stabilized Clay 1 is less than remolded Clay 1 because the hydration and cation exchange reactions induce the clay particle to larger lumps.

Fig.6 (b) shows the micrograph of the 20% lime stabilized Clay 2. It indicates clay aggregates at several places of micrograph. The formation of well-formed aggregates is due to the reaction of the lime on the soil particle. In the case of 20% lime stabilized Clay 1 as shown in Fig.6 (a), the micrograph indicates an open fabric element. There are many pore spaces in the structure of soil no the bound bridge between the clusters that show no cementing products. Figs.7 (a), (b) and Figs.8 (a), (b) show the micrographs of cement stabilized Clay 1 and Clay 2 at curing time 7 days. The microstructure of stabilized clays is different from the remolded clays. The formation of cementing products, which is visible, induces the decrease in pore spaces.

Fig.9 (b) shows that the microstructure of lime stabilized Clay 2 at curing time 28 days is exactly different from the remolded Clay 2 (Fig.4 (b)). The stabilized Clay 2 shows the presence of the cementing products and the decrease in pore spaces. The micrograph of Clay 1, which was mixed with 20% lime at curing time 28 days, is shown in Fig.9 (a). There are many pore spaces in fabric of the stabilized Clay 1. Figs.9 (c) and (d) show the enlarged view of lime stabilized Clay 1 and Clay 2 as the same samples in Figs.9 (a) and (b), respectively. They clearly present that the aggregate in fabric system of lime stabilized Clay 2 is denser than lime stabilized Clay 1.

# The hydraulic conductivity

The hydraulic conductivity of the remolded Clay 1 and Clay 2, under a vertical effective stress of 5 kPa, has been measured as  $2.56 \times 10^{-7}$  and  $2.09 \times 10^{-7}$  m/s. At the same vertical effective stress of 5 kPa, the void ratio of Clay 1 and Clay 2 are 3.65 and 3.55, respectively. The void ratio of Clay 1 is higher than Clay 2 at the same vertical effective stress. On the other hand, at the same void ratio or water content, vertical effective stress of Clay 1 is higher than Clay 2 because liquidity index of Clay 1 is lower than Clay 2. A hydraulic conductivity test is presented as function of the void ratio.



Fig.7 (a) Clay 1 mixed with 10% cement at curing time 7 days



Fig.7 (b) Clay 2 mixed with 10% cement at curing time 7 days



Fig.8 (a) Clay 1 mixed with 20% cement at curing time 7 days



Fig.8 (b) Clay 2 mixed with 20% cement at curing time 7 days



Fig.9 (a) Clay 1 mixed with 20% lime at curing time 28 days



Fig.9 (b) Clay 2 mixed with 10% lime at curing time 28 days



Fig.9 (c) Clay 1 mixed with 20% lime at curing time 28 days



Fig.9 (d) Clay 2 mixed with 10% lime at curing time 28 days



Fig.10 (a)Plot of e-logov' and e-log k relations in remolded and lime stabilized Clay 1



Fig.10 (b)Plot of e-logov' and e-log k relations in remolded and cement stabilized Clay 1

The permeability of Clay 1 is higher than Clay 2 at the same vertical effective stress. The permeability is the parameter that directly relates to the pore spaces in clay fabric. In the Fig.10 (a), the permeability of remolded Clay 1 and lime-stabilized Clay 1 are almost the same because the microstructure does not change after being mixed with lime. The pore spaces in fabric are the same because the cementing products are not formed.

Fig.11(a) shows the permeability of lime-stabilized Clay 2. The coefficient of permeability, k decreases with the lime content increase. The pore spaces are decreased because of the formation of cementing products as shown in the micrograph (Figs.5 (b), 6 (b)), which agree well with the previous research (Locat et al.1996). Figs.10 (b) and 11(b) show the permeability of cement-stabilized Clay 1 and Clay 2. The permeability decreases with the increase in cement content. To study the cementation effect, Ariake clay mixed with 5% cement and cured 28 days was also investigated by Yamadera (1999). The result reports that the microstructure of the cement stabilized and remolded clays are not different. Their pore space is almost the same because the cement content is quite low. The formation of cementing products is not adequate to affect the pore spaces. In this study, the ce-



Fig 11 (a) Plot of e-log $\sigma_v$ ' and e-log k relations in remolded and lime stabilized Clay 2



Fig 11 (b) Plot of e-log $\sigma_v$ ' and e-log k relations in remolded and cement stabilized Clay 2

ment content is high that is adequate to investigate the formation of cementing products in the fabric.

# Strength and compressibility

The strength of Clay 1, which is mixed with 20% lime and cured 28 days, is very low as about 5 kPa. Fig.12 (b) shows the strength development of lime and cement stabilized Clay 2. The lime is more effective than cement to stabilize Clay 2 because the strength of lime stabilized Clay 2 is higher than cement stabilized Clay 2 at the same admixture content. The strengths of lime and cement stabilized Clay 2 increase with the admixture contents and curing time. The strength development of cement stabilized clay at 7 to 14 days is faster than 14 to 28 days because the strength of cement stabilized clay is mainly achieved by hydration that mobilized at initial stage. It is different from lime-stabilized clay because it derives from pozzolanic reaction, which is a kind of slow reaction. The strengths of 20% cement stabilized Clay 2 and Clay 1 at curing time 28 days are 1400 and 800 kPa, respectively (Figs.12 (a), (b)). It shows that the lime does not only fail to stabilize Clay 1 but the strength of cement stabilized Clay 1 is also low.

Fig.13 (a) shows that the compression curve of the remolded Clay 1 is a straight line. After Clay 1 is mixed with 10 and 20% lime contents, the yield stresses,  $\sigma_y$  are 10 and 20 kPa, respectively. The yield stresses of Clay 1, which is mixed with 10 and 20% cement contents, are very low as about 50 and 500 kPa. The yield stresses of 10% cement and lime stabilized Clay 2 are 200 and 900, respectively (Fig.13 (b)). The results of unconfined compression and oedometer tests demonstrate that the lime fails to stabilize Clay 1 because it cannot induce both the strength and the yield stress after Clay 1 is mixed with lime at the curing period.

The strength development of stabilized clays agrees well with the microstructure investigation from the previous part. The strength development relates to the microstructure, which is fabric and cementation. The strength of lime stabilized Clay 1 is very low then cementing products are invisible. The strength of lime and cement stabilized Clay 2 increase with curing time and admixture



Fig.13(a) One-dimensional compression curves of lime/ cement stabilized Clay 1

Fig.13(b) One-dimensional compression curves of lime/ cement stabilized Clay 2

contents then the fabric of stabilized Clay 2 and remolded Clay 2 is exactly different. The stabilized Clay 2 clearly shows the presence of the cementing products.

# **Retarding factors**

The type of clay has been found to be a major factor in lime stabilized clay. Montmorillonite and kaolinite clay respond better to lime than illite and chlorite clay. Soil rich in halloysite clay develops lower strength than any other types (Mateos,1964). The study shows that the clay mineral of Clay 1 is not the major factor for retarding strength of lime stabilized clay because this clay does not contain high content of halloysite.

Miura et al.(1988) reported that the lime fails to stabilized Ariake clay that contains organic matter more than 6 percent. As shown in Table 1, Clay 1 contains organic matter only 1 percent. The strength of lime stabilized Clay 1 is still very low although Clay 1 was treated with Hydrogen peroxide ( $H_2O_2$ ) to eliminate the organic matter before being mixed with 20% lime. It means that the organic matter does not affect the strength development of lime stabilized Clay 1. It agrees with the previous researches that some kinds of organic matter has no influence on the strength of the stabilized clay (Tremblay et al. 2000).

Fig.14 (a) shows the compression curves of 20% lime stabilized clay that are different in Treated Clay 1 contents at curing time 7 days. The yield stress of lime stabilized clays increase with Treated Clay 1 contents. The yield stress of lime stabilized Treated Clay 1 is 200 times of lime stabilized Clay 1. It means that sulfide has high influence in yield stress of lime stabilized Clay 1. The yield stress of the lime stabilized clay decreases with the increase in sulfide contents.

Fig.14 (b) also shows the one-dimensional compression curves of 20% cement stabilized clays that are different in Treated Clay 1 contents. The yield stress of cement stabilized Treated Clay 1 is higher than cement stabilized Clay 1. It means that the yield stress of cement stabilized soft clay is reduced as the sulfide content increases. The sulfide has the higher effect, which is strength reduction, on the lime stabilized clay than the cement stabilized clay. The sulfide prevents the formation of cementing products because the sulfide obstructs the dissolving of silica (SiO<sub>2</sub>) and alumina (Al<sub>2</sub> O<sub>3</sub>) in the clay. The sulfide has a detrimental effect on the strength mobilization of lime stabilized soft clay by retarding the pozzolanic reaction.

Portland cement contains tricalcium silicate ( $C_3S$ ), dicalcium silicate ( $C_2S$ ), tricalcium aluminate ( $C_3A$ ) and a solid solution described as tetracalcium alumino-ferrite ( $C_4A$ ) (Lea,1956). These four main constituents are major strength producing compounds. When the pore water of the soil encounters with the cement, the major cementing products are hydrated calcium silicate (CSH), hydrated calcium aluminates (CAH) and hydrated lime (Ca(OH)<sub>2</sub>), occurs. The cement stabilized clay also induces pozzolanic reaction because the Ca(OH)<sub>2</sub> is produced from the hydration reaction. To understand the mechanism of lime and cement stabilized clay, the schematic diagram is illustrated in Fig.15 that is modified from Saitoh et al.(1985). It shows that the reaction of lime stabilized clay is one section of the reaction of cement stabilized clay. The sulfide has a detrimental effect on the strength mobilization of cement stabilized soft clay by obstructing of the pozzolanic reaction. Even though the strength of cement stabilized sulfide clay can be obtained from the products of hydration.



Fig.14(a)One-dimensional compression curves of 20% lime stabilized Clay in various Clay 1 contents



Fig.14(b) One-dimensional compression curves of 20% cement stabilized Clay in various Clay 1 contents

The strength of lime stabilized sulfide clay is extremely low because the strength is mainly obtained by pozzolanic reaction.



Fig.15 Schematic diagram of lime and cement stabilized clay

# CONCLUSIONS

The main conclusions from this study are the following.

1 The micrograph clearly shows that the fabric of stabilized clay and remolded clay is exactly different. The formation of cementing products is clearly confirmed by the micrograph. Strengths of both stabilized clays increase with curing time and admixture contents. In the case of lime stabilized clay, which contains high sulfide content, cementing products are invisible from the micrograph and the strength is extremely low.

2 The decrease in permeability relates to the pore spaces in the fabric. In the case of the stabilized non-sulfide clay, the formation of cementing products induces the decrease in pore spaces then the permeability decreases. The same permeability of remolded clay and lime stabilized clay, which contains high sulfide content, are shown. It means that their fabric is of the same pattern.

3 The liquid limit can be related to the change of microstructure in stabilized clay. The higher liquid limit relates to the formation of cementing products, thus induces the higher water holding capacity.

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