INVESTIGATIONS ON REACTION PRODUCTS IN SOIL CEMENT

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ABSTRACT : Cement stabilization has been widely used to improve soft soils and grounds. Many researches have focused on study of the properties of the stabilized soil. However, more researches have to be done in order to elucidate how soil properties have been improved. The main objective of this research was therefore to investigate major hydration products which contribute strength development of soil cement by using X-ray diffractometer (XRD). In addition, correlations between reaction products and the developed strengths were also observed. A clayey soil with moderate moisture content was used in this study. Ordinary Portland cement (Type I) was mixed with the soil at a mix proportion of 200 kg/m³. Cylindrical specimens were prepared for unconfined compression strength tests at curing times of 3, 7, 14, 28 and 90 days, respectively. Subsequently, X-ray diffraction (XRD) analysis was performed after strength tests. Experimental results showed that strengths of the soil significantly increased with curing time, especially at short term (before 28 days) while, strengths slightly changed at long term. This investigation elucidated that increase in strength was attributed to the cement hydration within soil mass, resulting in formation of reaction products as analyzed by XRD. It was also found that unconfined compressive strengths were proportionally increased with amount of the major reaction products such as calcium silicate hydrate (CSH) and ettringite.

KEYWORDS : Soil cement, Unconfined compressive strength (UCS), Calcium silicate hydrate (CSH), Ettringite X-ray diffraction analysis (XRD)

1. Introduction

For many decades, engineers and researchers have attempted to solve problems posed by various types of soft ground. Constructions on such grounds may encounter with unstabilities arisen from low shear strength, substantial total and differential settlement, excessive seepage and liquefaction. Therefore, various methods of ground improvement have been developed in order to improve such unfavorable properties.

The developed techniques are based on the basic concepts of ground improvement which include the effects of densification, cementation, reinforcement, and drainage. Among many successful projects, it has been reported that ground improvement method using cement and lime is suitable to improve soft clayey ground having high water content and high compressibility. The techniques such as deep cement mixing method and soil cement columns have been widely used in many countries as well as in Thailand recently (DOH and JICA, 1998, Ruenkrairergsa, et al., 2002).

As main objective of this study, the concept of chemical stabilization was introduced to improve strength of a soft marine clay. Ordinary Portland Cement (Type I) was stabilizer used in this study. In Thailand, there are many researches that have focused on study of the properties of stabilized soils. However, explanation on how properties of a soil cement such as strength is improved needs further elucidation. This study therefore attempted to clarify on how strengths were improved.

Experimentally, the approaches used in this study consisted of unconfined compressive strength test to

evaluate hardening effects and subsequent X-ray diffraction analysis (XRD) to investigate the main chemical compounds of materials and reaction products. Correlations between major reaction products and the developed strengths were also observed.

2. Materials and Methods

2.1 Materials

The clayey soil used in this study was sampled from a construction site at Klong Lat Pho, Samut Prakarn province. The ground water table level was about 1 m below ground surface. The disturbed soil was taken from a depth of 3.0 m., which was located below the weathered zone. Upon visual inspection, the soil had greenish to dark gray color, containing some organic fractions. Further data on their properties are given in Table 1. The soil can be classified as clay with high plasticity (CH) according to the Unified Soil Classification System.

Physical and engineering properties	Value
Liquid limit (%)	85.80
Plastic limit (%)	32.70
Plasticity index (%)	53.10
Shrinkage limit (%)	32.91
Wet unit weight (t/m^3)	1.65
Specific gravity	2.76
Natural moisture content (%)	63.53
Strength of untreated soil (ksc)	0.07-0.10
Permeability (cm/s)	6.71 x 10 ⁻⁶

Investigations on chemical compositions of the untreated soils were performed in accordance with ASTM C 323. The results showed that the soils were consist of 0.8 % Calcium oxide (CaO), 1.7 % Magnesium oxide (MgO), 1.0 % Sodium oxide (Na₂O) and 0.08 % Sulphate ion (SO₄²⁻). The remolded strength of untreated soil at its average natural moisture content (63.53%) was within a range of 0.07-0.10 ksc. Based on the test results, the soil was conformed to soft marine clay.

2.2 Specimen preparation and tests

Selection on appropriate stabilizer and mix proportion was done as suggested by previous researches (Nontananandh and Amornfa, 2002). It has been recommended that cement had beneficial effects to improve foundation of soft Bangkok clay, i.e. ground improvement by cement column. Suitable cement content was within a range of 80-200 kg/m³ based on required design strength of each project (DOH and JICA 1998). Therefore, Ordinary Portland Cement (Type I) at a 200 kg/m³ was used in this study to stabilize the soft marine clay. In addition, a water to stabilizer ratio of 0.80 was used to produce soil-cement mixture.

Mixing and preparation of specimens was performed carefully to produce a homogeneous soil-cement mixture with a constant initial density of all specimens. After mixing, cylindrical specimens of 5 cm diameter by 10 cm long were made for strength tests. After de-molding, the specimens were sealed tightly in plastic sheets to prevent loss of moisture due to surface evaporation and then cured for periods of 3, 7, 14, 28 and 90 days before strength tests. Unconfined Compression Strength Test was performed in accordance with ASTM D 2166-91.

Investigations on reaction products such as calcium silicate hydrate (CSH), ettringite and calcium hydroxide (Ca(OH)₂) were performed on both cement pastes and the failures of specimens after strength test. XRD tests were done by using a Philips X'Pert Diffractometer with an input energy of 40 kV and 30 mA and a scanning speed of 2 degrees/min. This study identified CSH, ettringite and Ca(OH)₂ at d-spacings of 3.02 Å, 3.88 Å and 2.62 Å respectively where strong reflections were prominent and did not overlap with other phases.

3. Results and Discussions

3.1 Strength and deformation characteristics

The characteristic curves showing the development of strength of soil cement against curing time is presented in Figure 1. Experimental results showed that unconfined compressive strengths (UCS) were significantly increased when compared with strength of the untreated soil. Strengths significantly increased during the first two weeks, while, at long term, strength slightly increased. It was revealed that the stabilized soils could obtain preferable strengths and therefore showed potential uses for ground improvement purposes such as soil cement column or subbase materials for roads.

Hardening effect established cementing characteristic, resulting in an increase in modulus of elasticity (E_{50}). As observed in Figure 2, E_{50} markedly improved at short

term, while, slightly increased at long term. This result was similar to strength development. As shown in Figure 3, strengths and E_{50} exhibited a linear correlation as fitted using the method of least squares. An increase in E_{50} implied that the soils with high plasticity were changed into more rigidity material. After 28 days, the soil cement could obtain E_{50} approximately 1,600 ksc, which was equivalent to dense sand.



Figure 1 UCS of soil cement with curing time



Figure 2 E₅₀ of soil cement and curing time



Figure 3 UCS and E₅₀ of soil cement

3.2) XRD patterns and reaction products in relation to strength development

The XRD pattern of the untreated soil, as illustrated in Figure 4, indicated that the soil was composed silica in the form of quartz, montmorillonite, illite, and kaolinite as dominant minerals. Identification revealed that the untreated soil initially contained no cementing materials.



Figure 4 XRD pattern of untreated soil



Figure 5 Typical XRD pattern of cement paste (at 150 kg/m³, 3 days)



Figure 6 Typical XRD pattern of cement paste (at 150 kg/m³, 28 days)



Figure 7 CSH intensity of cement pastes

Preliminary tests on the XRD analysis of cement pastes, as illustrated in Figure 5 and Figure 6, indicated that CSH, $Ca(OH)_2$ and ettringite were major reaction products. It could be observed that the intensities of these reaction products increased with curing time.



Figure 8 Ettringite intensity of cement pastes



4000 Qt = Quartz 3500 Mont = Montmorilonite Ilt = Illite 3000 Kao = Kaolinit CSH = Calcium Sillicate Hydrate 2500 Et = Ettringite counts/s 2000 1500 CSH 1000 500 Mont 0 10 20 30 40 50 60 70 80 2Theta

Figure 10 XRD pattern of soil cement (90 days)

Consequently, the diffraction intensities of CSH and ettringite also increased with increase in cement content, as shown in Figure 7 and Figure 8. It could be observed that CSH and ettringite intensity markedly increased during the first two weeks, then slightly increased and became almost constant at long term. Higher reflections were obtained from the mixtures having relatively higher cement contents. When compared with CSH, ettringite was produced at relatively smaller intensity.

Typical XRD patterns of the cement stabilized soil at 3 and 90 days, as illustrated in Figure 9 and Figure 10 showed growths of major reaction products which could be identified as CSH and ettringite. However, in contrary to cement paste, Ca(OH)₂ could not be detected since the early stage. This result, therefore, revealed that calcium hydroxide dissolved rapidly as Ca²⁺ and (OH)⁻ into pore solutions after cement hydration, enhancing subsequent dissolution of some silicate and aluminate from clay minerals, and pozzolanic reaction at long term.



Figure 11 CSH intensity of soil cement against time



Figure 12 Ettringite intensity of soil cement against time

Figure 11 and Figure 12 illustrate X-ray intensities of CSH and ettringite with curing time. In similar to the characteristic curves of strength development and cement pastes, CSH and ettringite intensity characteristic curves of the cement stabilized soil markedly increased during the first two weeks, then slightly increased and became almost constant at long term. In addition, as illustrated in Figure 13 and Figure 14, strengths were increased proportionally with amounts of CSH and ettringite that formed. It could therefore be concluded that these reaction products mainly contribute to strength development of the cement stabilized soils.

It was assumed that initial hardening effect was additionally enhanced due to formation of reaction product such as ettringite. Essential role of ettringite (3CaO Al₂O₃ 3CaSO₄ 32H₂O) was attributed to the fact that large amount of water was initially combined in its crystals, resulting in significant decrease in moisture content at the early age. Extracting water that existed in the pore spaces by ettringite provided a reduced water to cement ratio that aided further hardening. In addition, CSH were thus formed simultaneously at the beginning course of reaction. Significant increase in strength of the stabilized soil was due to growth and hardening phases of CSH that bound soil particles together. Hardened and denser structure was gradually formed, resulting in a substantial increase in soil strength with progressive of curing time.

Supplement explanation on strength development due to subsequent changes on soil structures should be further elucidated. This could be accomplished by using the advantage of Scanning Electron Microscopic observation (SEM) of the cement stabilized soil. The results were reported in a separate paper.



Figure 13 UCS and CSH intensity of soil cement



Figure 14 UCS and ettringite intensity of soil cement

4. Conclusion

Based on the results of this study, it could be concluded that strengths of a soft marine clay significantly increased when mixed with cement at a suitable content. It was found that growths of CSH and ettringite with curing time were similar to strength characteristic curves. Strengths were increased proportionally with amounts of the major hydration products such as CSH and ettringite that formed. It was therefore concluded that hardening effects of the cement-stabilized marine clay was substantially influenced by the formation of the major reaction products such as calcium silicate hydrate (CSH) and ettringite.

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6. Reference

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