Effect of Cement Content on Strength and Microstructure of Cement Stabilized Clay

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Abstract : This paper presents the effects of cement content on the strength development of cement stabilized clay. A qualitative and quantitative study on the microstructure of the stabilized clay was carried out using scanning electron microscope, and mercury intrusion pore size distribution measurements. It is found that the relationship between strength and cement content at a particular water content is divided into three zones: active, inert, and decline zones. The difference in the microstructure of the stabilized samples in each zone is realized by the hydration products and pore size distribution. Initially, strength increases markedly with cement content and becomes inert in the second zone where the maximum strength occurs. The samples of highest strength possess the highest amount of hydration products and the lowest total pore volume. In the decline zone, the hydration products are insignificantly noticed from SEM photos and the total pore volume is larger than that of the other two zones. It is because the water is not significantly for complete hydration and compaction. Thus the large clay-cement clusters with high resistance to compaction are formed, resulting in large pore space.

1. Introduction

Soil in northeast Thailand generally consists of two layers. Upper layer (varying from 0-3 m thickness) was wind-blown and deposited for several decades. It is clayey sand or silty clay with low to moderate strength ($12 < N < 20$, where N is standard penetration number). This upper soil is a problematic soil, which is sensitive to change of water content [1]. The laboratory and field investigations on its collapse behavior due to wetting are illustrated [2-4]. The lower layer is a residual soil, weathered from claystone, consisting of clay, silt and sand [5]. It possesses very high strength (generally $N > 30$) and very low compressibility. One of the extensively used ground improvement techniques for the upper soil is to compact the in-situ soil (in relatively dry state) mixed with cement. This technique is economical because cement is readily available at reasonable cost in Thailand. Moreover, adequate strength can be achieved in a short time.

The stabilization is begun with mixing of the soil in a relatively dry state with cement and water specified for compaction. The soil, in the presence of moisture and cementing agent gets transformed into modified soil i.e, grouping of particles takes place due to physico-chemical interactions with soil~cement~water interactions. Since these are at particle level, it is not possible to get a homogeneous mass, which can exhibit the desired levels of strength. The compaction effort is needed to remove air from the soil (to increase degree of saturation) and to make soil particles slip over each other and move into a densely packed state. At this state, the soil particles can be well welded by chemical (cementation) bond (in which the bond strength is controlled by molding water content, cement content and curing time) and become an engineering material.

Models of the microstructure of fine-grained soils have been advanced since 1953

RECEIVED 7 August, 2007
ACCEPTED 6 December, 2007

by geotechnical engineers in order to help in understanding their engineering behavior. Lambe's model is the first conceptual model, which considers the clay particle as the single platelet. Since Lambe developed his theory, there have been significant improvements in the techniques of observing microstructures, leading towards complete description of the microstructure of fine-grained soils in relation to their engineering behavior such as the works reported by Gillott [6] and Collins and McFown [7]. Aylmore and Quirk [8] and Olsen [9] have revealed that the basic element of the microstructure of the natural clay is not the single platelet but domains constituted of various platelets aggregated together.

Nagaraj et al. [10] have analyzed the pore size distribution of saturated clay and concluded that the volume of pores larger than 0.03 micron amounts to nearly 90 to 95% the total void volume of the soil. The pore smaller than 0.01 micron is the intra-aggregate pore which is in the stable clusters formed in different electrolytic environments during deposition and sediment formation. This pore volume accounts for only 3 to 5% the total pore volume. Since net force is likely to be attractive, these clusters are stable even in the absence of external loading. When such stable units are in close proximity in the range of separation distance of 0.01 to 0.03 micron, the net force of repulsion (*R-A*) between such units would create an osmotic suction on adjacent fluid equal in magnitude to the isotropic mean effective stress. This separation distance of 0.01 to 0.03 micron is referred to as inter-aggregate pore between two interacting aggregate (micro inter-aggregate pore). When pore fluid is subjected to such suction pressure at innumerable points, due to several pairs of interacting particle units, a large pore (larger than 0.03 micron) can be formed. This pore is designated as inter-aggregate large enclosed pore held within a group of clusters by surface tension (macro inter-aggregate pore).

Even though the investigations on the microstructure are available, they concentrated mainly on natural clay. This paper aims to investigate the microstructural characteristics of clay stabilized by cement so as to explain the role of cement content on the strength development.

2. Experimental Investigation

Soil sample is silty clay collected from the campus of Suranaree University of Technology, Nakhon Ratchasima, Thailand at 3 meter depth. The soil is composed of 2% sand, 45% silt and 53% clay. Specific gravity of the soil is 2.74. The liquid and plastic limits are in the order of 74 and 27 percent, respectively. Based on the Unified Soil Classification System (USCS), the clay is classified as high plasticity (CH). During sampling, the groundwater disappears. Natural water content was 10 percent. The free swell test [11] shows that the clay is low swelling type with free swell ratio (FSR) of 1.0. The FSR is defined as the ratio of equilibrium sediment volume of 10 g of oven-dried soil passing a 425 mm sieve in distilled water (V_d) to that in kerosene (V_k). This method was employed since it is a simple methodology to obtain a approximate and fairly satisfactory prediction of the dominant clay mineralogy of soil [12].

The clay was passed through a 16-mm sieve to remove coarser particles. It was air-dried for at least three days and then water content was adjusted for compaction test. At least five compaction points were generated. The compaction was carried out according to ASTM D 1557 in a standard 100-mm diameter mold for modified Proctor energies (2693.3 kJ/m^3). The compaction characteristics (optimum water content, *OWC*, and maximum dry unit weight, $\gamma_{d\max}$) are 17.2% and 17.3 kN/m^3.

Having obtained the compaction curves, the air-dried clay was thoroughly mixed with ordinary (Type I) Portland cement at different cement content from 3 to 45%. The clay-cement powder was then thoroughly mixed with water to attain 17% water content and compacted. Specific gravity of the cement is 3.15. Chemical composition and grain size distribution of the cement compared with those of silty clay are shown in Table 1 and Figure 1, respectively. Grain size distribution of the cement is obtained from the laser particle size analysis. It is found that the particle of the silty clay is much smaller than that of the cement. SiO_2 and Al_2O_3, which are the main elements for pozzalanic reaction, are almost the same for both the cement and the silty clay. The summation amount of these two major components in the silty clay is very low (less than 30%), therefore the silty clay can be considered as a non-particulate material.

Table 1 Chemical composition of Type I Portland cement and silty clay.

Chemical composition (%)	Cement	Silty clay
SiO_2	20.9	20.1
Al_2O_3	4.76	7.55
Fe_2O_3	3.41	32.89
CaO	65.41	26.15
MgO	1.25	0.47
SO_3	2.71	4.92
Na_2O	0.24	ND
K_2O	0.35	3.17
LOI	0.96	3.44

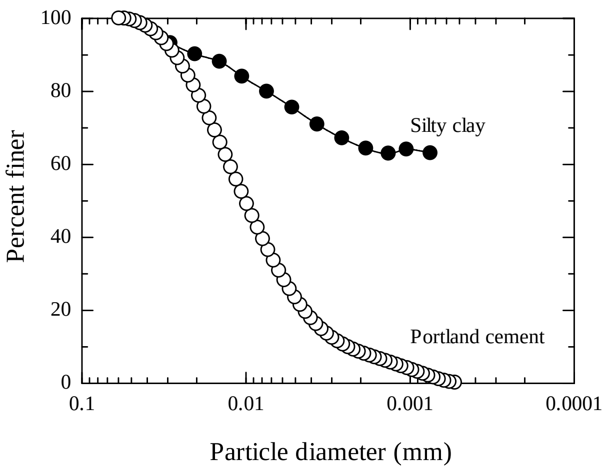


Figure 1 Grain size distributions of Portland cement and silty clay.

After 24 hours, the cement stabilized samples were dismantled, wrapped in vinyl bags and stored in a humidity chamber of constant temperature (25±2°C). Unconfined compression test was run on the samples after 7 days of curing. The rate of vertical displacement was fixed at 1 mm/min. For each curing time, and each combination of water content and cement content, at least three samples were tested under the same condition to check for consistency of the test. In most cases, the results under the same testing condition were reproducible.

The microstructure of the cement stabilized clay is investigated by the scanning electron microscope (SEM), and mercury intrusion porosimetry (MIP). The cement stabilized samples at the required curing times were broken from the center into small fragments. The SEM samples were frozen at -195°C by immersion in liquid nitrogen for 5 minutes and evacuated at a pressure of 0.5 Pa at -40°C for 5 days [13 and 14]. All samples were coated with gold before using SEM (JOEL JSM-6400) analysis.

Measurement on pore size distribution of the stabilized samples was carried out using mercury intrusion porosimeter (MIP) with a pressure range from 0 to 288 MPa, capable of measuring pore size diameter down to 5.7 nm (0.0057 micron). The MIP samples were obtained by carefully breaking the stabilized samples with a chisel. The representative samples of 3-6 mm pieces weighing between 1.0-1.5 g were taken from the middle of the stabilized samples. Hydration of the samples was stopped by freezing and drying, as prepared in the SEM examination. Mercury porosimetry is expressed by the Washburn equation [15]. A constant contact angle (θ) of 140° and a constant surface tension of mercury (γ) of 480 dynes/cm were used for pore size calculation as suggested by Eq.(1)

$$D = -\left(4\gamma \cos\theta\right)/P \qquad (1)$$

where D is the pore diameter (micron) and P is the applied pressure (MPa).

3. Test Results and Discussion

Influence of cement content on the strength development of the cement stabilized clay at a particular initial water content (17%), compaction energy (2693.3 kJ/m^3), and curing time (7 days) is presented in Figure 2.

The strength increase can be classified into three zones. As the cement content increases, the cement content per grain contact point increases and upon hardening, imparts a commensurate amount of bonding at the contact points. This zone is designated as active zone. Beyond this zone, the strength development slows down with gradual increase. The incremental gradient becomes nearly zero and does not make any further significant improvement. The zone is referred to as the inert zone (cement content ranging from 11-30%). The strength decrease appears when cement content is higher than 30%. This zone is identified as the decline zone.

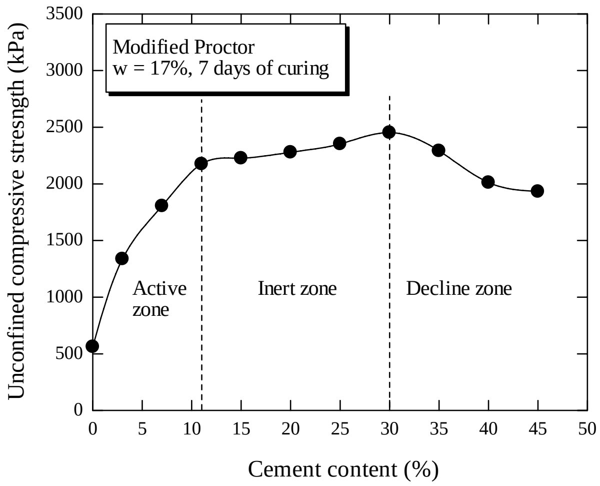
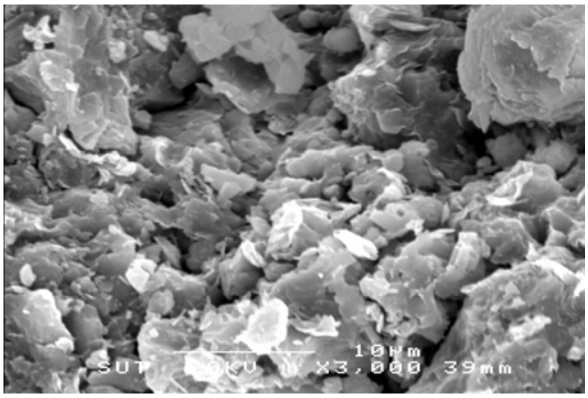


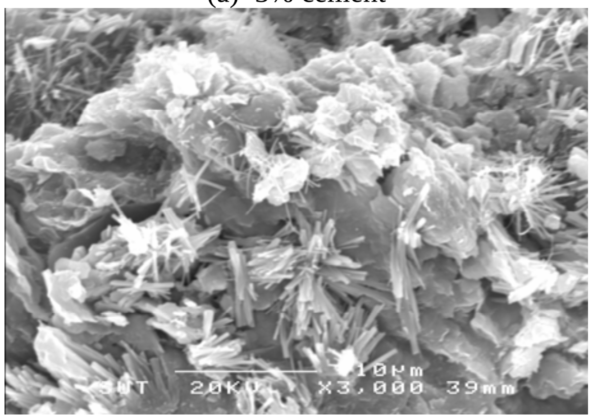
Figure 2 Improvement zones.

Figures 3 through 5 show the SEM photos of the cemented samples compacted at water content of 17% for active, inert and decline zones after 7 days of curing. SEM photo of 3% cement sample (Figure 3a) clearly shows the clay platelet and clay clusters with insignificant hydration products since the input of cement is small compared to the soil mass. Ettringite is clearly seen in the pore space and significantly increases with cement content (Figures 3b and 3c) up to C = 11%, hence strength increase.

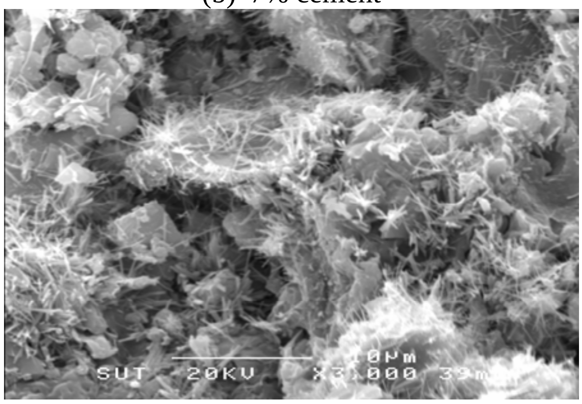
For inert zone (Figures 4a to 4d), the presence of ettringite is almost the same amount for all cement contents. This results in insignificant increase in strength. For decline zone (Figures 5a to 5c), ettringite decreases with cement, hence strength decrease. This might be because water is not enough for hydration. It is thus concluded that for a particular water content and compaction energy, the input of cement excess active zone is useless. In the active zone, water is enough for hydration and the cement gel could cover the clusters. When the gel hardens, the cementation welds the clusters and fills up the pore space.



(a) 3% cement

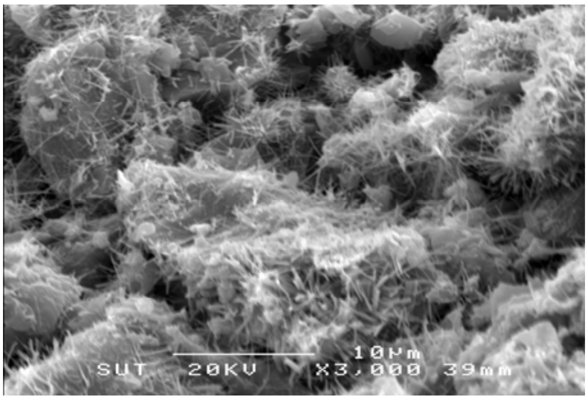


(b) 7% cement

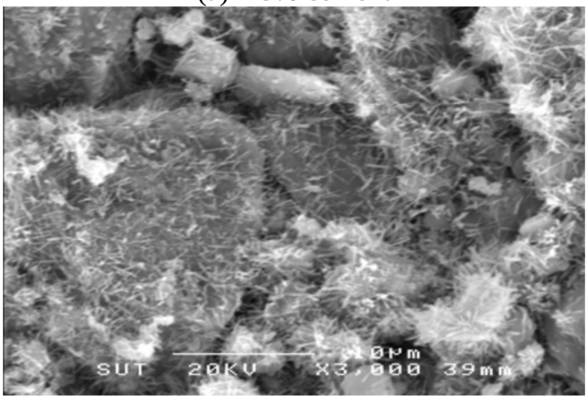


(c) 11% cement

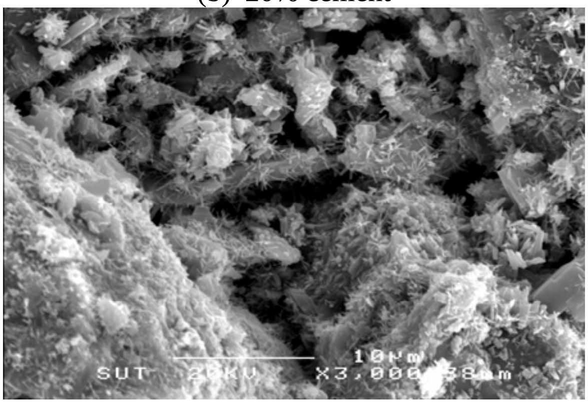
Figure 3 SEM photos of cement stabilized clay in active zone after 7 days of curing.



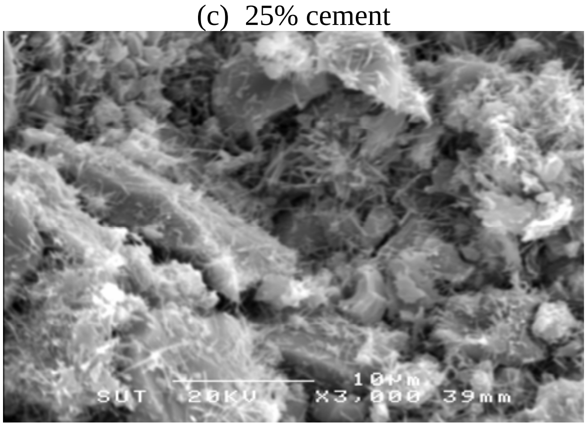
(a) 15% cement



(b) 20% cement

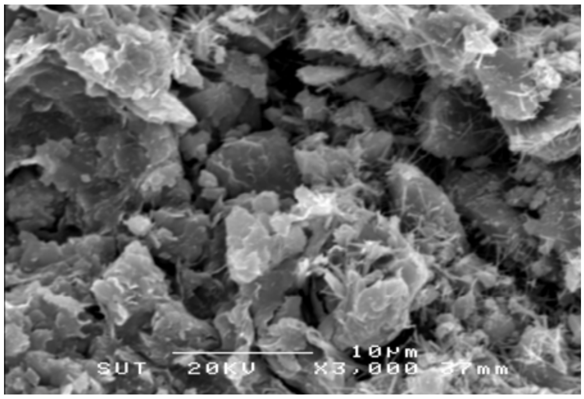


(c) 25% cement

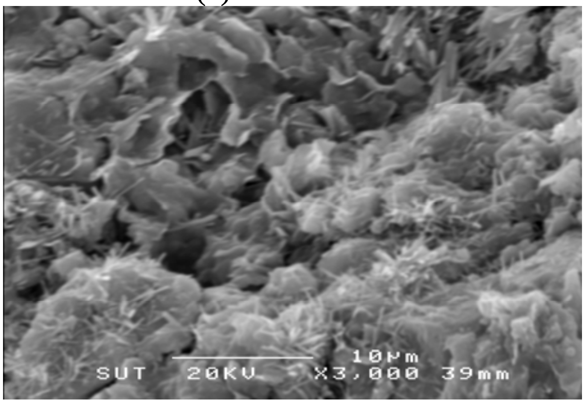


(d) 30% cement

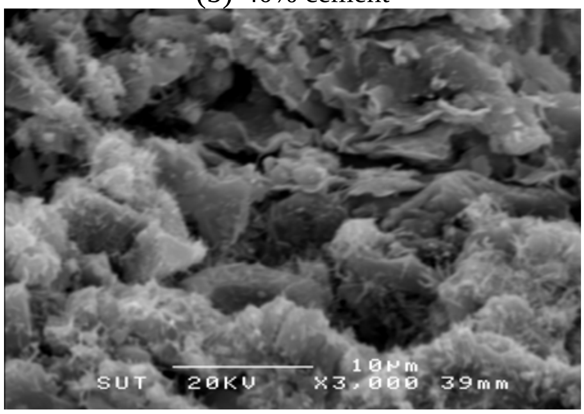
Figure 4 SEM photos of cement stabilized clay in inert zone after 7 days of curing.



(a) 35% cement



(b) 40% cement



(c) 45% cement

Figure 5 SEM photos of cement stabilized clay in decline zone after 7 days of curing.

The pore size distribution of the cement stabilized clay for different cement contents is illustrated in Figures 6 and 7. The number in the bracket of Figure 7 shows percentage of each range of the pore volume. It is shown that the pores larger than 0.03 micron (macro inter-aggregate pore) amount to nearly 90% the total void volume and the pores smaller than 0.01 micron account for only 3 to 5% total pore volume. This is in agreement with the pore size distribution of saturated clays analyzed by Nagaraj et al. [10]. As such, it is possible to state that the relative pore volume remains almost the same, irrespectively of compaction and the compaction only reduces the total pore volume.

The maximum population of the pore (dominant pore) is about 0.2-0.4 micron. The pores larger than 10 micron and smaller than 0.01 micron (intra-aggregate pore) have almost no change with cement content. Thus, it is logical to mention that the hydration products filled up the 10-0.01 micron pores. For C = 3-15%, the incremental pore volume for 0.10-0.01 micron pores reduces significantly with cement, resulting in the decrease in total pore volume. The incremental pore volume at the maximum population (0.2-0.4 micron) increases with cement content whereas total pore volume decreases. In inert zone, incremental pore volume at the maximum population (0.2-0.4 micron) increases with small reduction in total pore. This implies that in both active and inert zones, large pores are filled up by cementing products hence the small pores increase. In the decline zone, incremental pore volume at the maximum population (0.2-0.4 micron) decreases with cement while total pore increases. This shows that the increase in cement induces the large pore and hence the total pore volume. This is because the increase in cement reduces the water in the clay leading to the formation of large clay-cement clusters. These clusters are hard with high resistance to compaction energy. As such, the presence of large pore (0.1-1 micron) is remarkable.

Moreover, it is shown from Figure 6b that at a particular pore diameter, the total pore reduces with increasing cement content up to 30% cement and tends to increase beyond this value. The 3% cement sample (upper curve) has the largest cumulative pore diameter (n = 30.35%) whereas the 30% cement sample (lowest curve) has smallest cumulative pore diameter (n = 28.94%). The lowest cumulative pore diameter associates with the highest strength. The pore size distribution curves of 15% and 30% cement samples are similar since both samples exhibit almost the same strength.

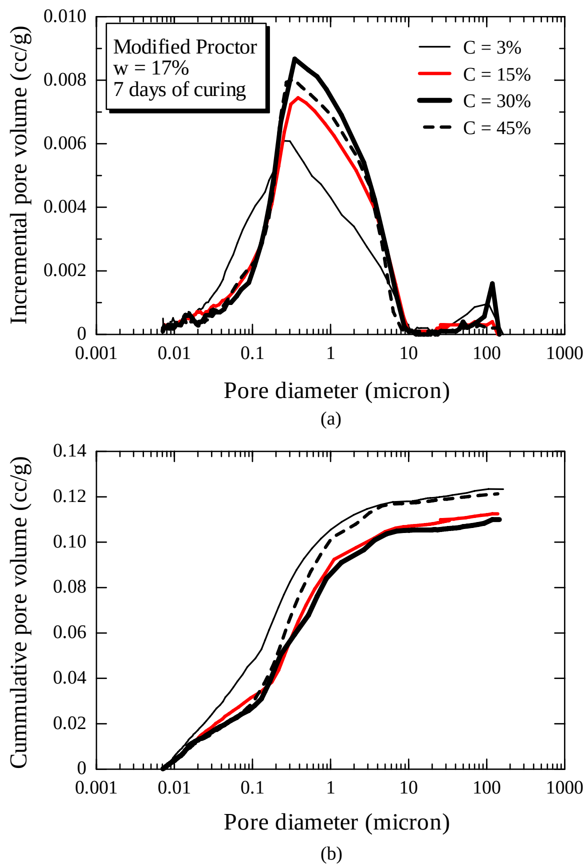


Figure 6 Effect of cement content on pore size distribution.

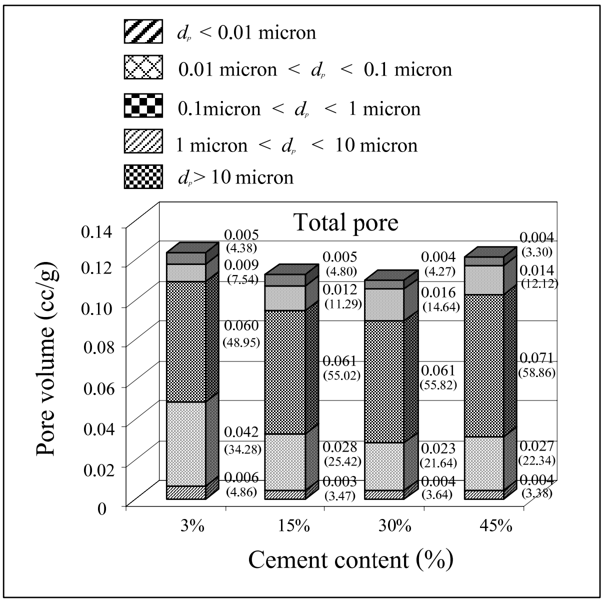


Figure 7 Relationship between pore volume and cement content after 7 days of curing.

4. Conclusions

This paper mainly deals with the analysis of strength development in cement stabilized clay based on the microstructural consideration. The conclusions can be drawn as follows.

1. At a particular water content, the cement stabilization is divided into three zones: active, inert and decline. In the active zone, as the cement content increases, the cement content per grain contact point increases and upon hardening, imparts a commensurate amount of bonding at the contact points. In the inert zone, the strength development slows down with gradual increase. The strength decrease appears in the decline zone due to insignificant water for compaction and hydration. In practice, the stabilization should be done in the active zone.
2. SEM photos of the cemented clay show that the hydration products remarkly increase with cement in the active zone. The amount of hydration products is insignificantly changed with cement in the inert zone. In the decline zone, the hydration products tend to decrease with cement content.
3. From the pore size distribution analysis, it is found the dominant pore is 0.2-0.4 micron. In both active and inert zones, large pore is filled up by cementing products thus the small pore increases. In the decline zone, incremental pore volume at the maximum population (0.2-0.4 micron) decreases with cement while total pore increases. This shows that the increase in cement induces the large clusters and large pore space and hence the total pore volume. This is because the increase in cement reduces the water in the clay leading to the formation of large clay-cement clusters.

5. Acknowledgement

The authors would like to acknowledge the financial support provided by the Thailand Research Fund and the Commission on Higher Education under contract MRG5080127.

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