Laboratory TDR monitoring of physico-chemical process in lime kiln dust stabilized clayey soils

CHEN Ren-peng[1]，DAITA R K[2]，DRNEVICH V P[2]，KIM D H[3]

(1. Geotechnical Engineering Institute, Zhejiang University, Hangzhou 310027, China; 2. Purdue University, School of Civil Engineering, West Lafayette, IN 47906, USA; 3. Indiana Department of Transportation Research Division, West Lafayette, IN 47906, USA)

Abstract: By-product materials like lime kiln dust (LKD) and cement kiln dust (CKD) begin to be used in the highways and airfield subgrades for improving natural soils to make them suitable construction materials. Most often, dry density and water content are measured in the field for the quality control purposes. Due to the complex physico-chemical reactions in the mixture, the maximum dry density and the optimum water content of the stabilized soils are always quite different from those of natural soil, which make it difficult to evaluate the quality of the stabilized soil. The physico-chemical process in LKD stabilized soil resulted in the change of the electromagnetic properties, which could be monitored by robust, automated, time domain reflectometry (TDR) technology. LKD stabilized orchard clay with different dosages of LKD and water content were compacted in standard molds. The electrical conductivity and apparent dielectric constant were monitored by TDR to investigate the physico-chemical processes. Needle penetrometer tests were also done on replicate samples at different curing time. It was found that the variation of electrical conductivity and apparent dielectric constant with time was an indicator of short-term cation exchange and agglomeration and long-term pozzolanic reactions. The decrease of electrical conductivity with time coincided with the increase of the penetrometer resistance. The initial (less than one day) reduction of conductivity was strongly related to the dosage of LKD, while the long term reduction was related to the strength development. The paper presented the potential applications of TDR technology to the LKD stabilized soil in the field quality control.

Key words: lime stabilized soil; lime kiln dust; TDR; physicochemical process; pozzolanic reaction; cation exchange

室内 TDR 试验监测石灰矿渣加固粘性土的物理化学反应过程

陈仁朋[1]，DAITA R K[2]，DRNEVICH V P[2]，KIM D H[3]

（1. 浙江大学建筑工程学院，浙江 杭州 310027；2. 美国普度大学土木学院，美国；3. 印第安那交通局研究部，美国）

摘　要： 石灰矿渣和水泥矿渣常用于加固道路基层的粘性土。目前现场一般采用未加固土的最大干密度和最优含水率来评价加固土的压实施工质量。但是粘性土掺加了石灰后，发生了复杂的物理化学反应，使得其压实特性与未加固土有很大的不同。采用时域反射计监测石灰加固土内的物理化学反应过程。试验时将不同石灰矿渣掺和量和含水率的 Orchard 粘土通过标准击实试验夯实于击实筒中，用 TDR 监测其中的物理化学反应，针式贯入仪监测强度的增长。试验发现，电导率和介电常数的变化反应了土中的离子交换和硬化反应的过程。初始电导率（1 d 之内）的减小揭示了石灰矿渣的掺和量的大小，而长期电导率的减小揭示了强度的增长。试验表明，时域反射计技术可用于评价石灰加固土的施工质量。

关键词： 石灰土；石灰矿渣；时域反射计；物理化学反应；硬化反应；离子交换

0　Introduction

Chemical stabilization is widely used in geotechnical engineering practice for improving the engineering properties of geo-materials. Extensive research and field applications show that the use of chemical stabilization can significantly improve the engineering properties of the soils. For the past century, the most widely used stabilizers are lime for clayey soils

and cement for both clayey and sandy soils. Recently, lime kiln dust (LKD) and cement kiln dust (CKD), the by-products of lime and cement industries, are being utilized as soil stabilizers. Physicochemical processes of stabilized soil have been extensively studied by previous researchers[1,2]. Microscopic technologies, for example SEM and X-ray diffraction analysis, are used to investigate the morphological changes and chemical products in stabilized soil. Atterberg Limits, shrinkage, swell-potential, leaching, resilient modulus, and strength are extensively studied engineering properties for stabilized soils.

The main physicochemical processes that occur upon addition of lime or lime kiln dust to soil are hydration of calcium oxide, cation exchange and agglomeration, and pozzolanic reaction. These processes take place at different rates and it is very difficult to directly monitor these processes. The physicochemical processes in the modified soil also change its electromagnetic properties with time. So it should be possible to monitor the electrical properties such as electrical conductivity and dielectric constant of stabilized soil to study the physicochemical process in the modified soil. Boardman et al. found that electrical conductivity of a lime modified soil decreases with time and concluded that in situ monitoring of stabilization process can be achieved by simple conductivity tests[3]. Yu and Drnevich showed that the electrical conductivity, as measured with Purdue time domain reflectrometry (TDR) apparatus, was an accurate and effective indicator of the progress of hydration in lime stabilized soil and the strength development of concrete[4].

The quality control of the stabilized soil is challenging as the problem involves a number of variables. For compacted natural soils, quality control is based on the dry density and water content specifications. For a stabilized soil, these properties are insufficient to achieve quality control and need additional information like strength, amount of lime, and depth and uniformity of mixing. The ability of TDR to measure the electrical conductivity and dielectric constant quickly and efficiently makes it a potential method for monitoring the physicochemical processes and the strength development in a stabilized soil.

This paper describes how the authors applied the TDR technology to monitor the electrical properties and use it as a means of studying the physicochemical changes of LKD-modified soils. The present findings show the potential applications of TDR technology to stabilized soils for field quality control.

1　The physico-chemical processes

Lime-based stabilizer in the form of, either quicklime (Calcium oxide (CaO)), hydrated lime (Ca(OH)$_2$), or lime kiln dust is primarily used to treat clayey soils. Lime reacts rapidly with the water in the soil and produces hydrated or slaked lime, which generates considerable heat, high concentration of calcium ions and hydroxyl ions:

$$CaO + H_2O = Ca^{2+} + 2OH^- + 65.3 \quad . \quad (1)$$
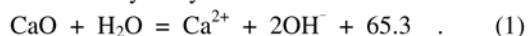
Generally the clayey soil particles are negatively charged due to the isomorphous substitution. The hydrated cations in conjunction with water molecules are attracted to the clay surface to neutralize the negative charge on clay cleavage. The negative surface and the distributed charge in the adjacent phase are together termed the diffuse double layer. The thickness of the diffuse double layer is related to the concentration and valence of the ions. The higher the concentration and the valence of the ions, the thinner is the diffuse double layer. The adsorbed cations within the double layer may readily be exchanged for other higher valence cations. A typical exchange series in ascending order of exchange preference is as follows[5]:

$$Na^+ < K^+ < Ca^{2+} < Mg^{2+} < Al^{3+} \quad . \quad (2)$$
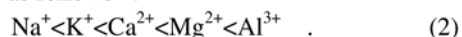
The lime-based stabilizer provides sufficient calcium ions in the pore water, which result in the replacement of the monovalent cations on the cleavage surface of clay by calcium ions. This process reduces the thickness of the double layer. The thickness of double layer determines the plasticity of the clay. An increase in electrolyte content of the pore water and adsorption of Ca^{2+} ions in the exchange process has been attributed to agglomeration of clay soil particles. Due to the cation exchange and agglomeration processes, the soil texture changes from that of a plastic, fine-grained material to like that of a more granular soil. The cation exchange and flocculation are together termed short-term reactions because these reactions occur within the first few hours of mixing of lime with the soil. Although cation exchange and flocculation are very quick in normal conditions, for lime stabilized soils diffusion of the Ca^{2+}

ions into the clay particles is slow and hence the reactions take longer time.

The high concentration of hydroxyl ions from the reaction shown in Eq. (1), generates a high pH environment in the soil. The solubility of silica and alumina within the clay is greatly increased. The solvated silica ions and aluminum ions will react with calcium ions to form the pozzolanic compounds: calcium-silicate-hydrate (C-S-H), and calcium-aluminate-hydrate (C-A-H), which have cementitious properties that promote the development of agglomerates[1]. These pozzolanic reactions occur over years, and dominate the long-term changes in soil stabilization. The pozzolanic reactions greatly increase the strength and stiffness and decrease the ductility of the stabilized soil.

The amount of lime or LKD necessary to achieve a desired level of stabilization is important. Excessive amounts of lime generally are detrimental to the modified properties of soil. Eades and Grim method is widely used to determine the lime requirement[6]. In this test about five samples of 25 g of air-dried soil solids are suspended in 100 ml of de-ionized water in plastic bottles. Variable amounts of lime ranging between 2% and 6% by weight of air-dried soil solids, in increments of 1%, are added to samples. The pH of each mixture is measured after an hour, and the minimum amount of lime that provides a pH of 12.4 is considered to be adequate to stabilize the soil under investigation. The dosage determined by the Eades and Grim method does not consider the long-term strength development.

2　Materials

The soil used in the experiments was Orchard clay, sampled from Orchard sub-division in West Lafayette, IN. The soil was tested for basic index properties of grain size distribution, Atterberg limits, compaction characteristics, pH, according to the corresponding ASTM specifications. The soil contains 28% sand, 47% silt and 25% clay. The liquid limit is 35%, and plastic limit is 16%. The maximum dry density is 19.75 kN/m^3 and optimum moister content(OMC) is 16.5% according to the standard proctor compaction.

The lime kiln dust was provided by Mount Carmel Sand and Gravel Company. The available lime in the LKD as provided by the supplier is 32.2%.

3　Sample preparation and measurement

Six samples of Orchard clay were prepared during this test program. Four samples were with 4% LKD of which two were at OMC, and two were at ± 2% of OMC. The remaining two samples were prepared with 2% and 6% LKD at their respective OMC. A summary of these tests is given in Table 1.

Table 1 Measurements profile

| Test ID	LKD percent/%	Premodification water content/%
A	2	17
B	4	15
C_1	4	17
C_2	4	17
D	4	19
E	6	17

Orchard clay was air-dried at room temperature and passed through a 4.75 mm (No.4) sieve. The sieved soil was mixed with target water content using tap water and sealed in plastic bags overnight in a humid room, which was maintained at a temperature of 10℃. The following day, the required amount of percent LKD calculated based on the weight of the soil solids was added and mixed using a mechanical mixer for five minutes. All the samples were compacted in three lifts with 25 blows per lift in a standard compaction mold, with a diameter of 10.16 cm and a height of 11.64 cm (without the collar) as shown in Fig. 1 and which follows ASTM D 698. For each test two replicate samples were prepared. One sample was used for measuring the electrical properties of the stabilized soil using the TDR apparatus and the replicate sample was used for determining the needle penetration resistance. All the compacted samples were covered by wax to prevent loss of moisture due to evaporation. For tests C_1 and C_2, the initial and final water contents of the soil in the mold were measured.

Electrical conductivity of a soil is dependent on its temperature and hence the temperature of the stabilized soil was monitored using a thermocouple and the variations in room temperature were monitored using a thermometer.
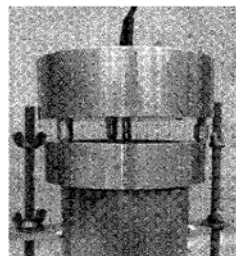
3.1　Measurement of electrical conductivity and dielectric constant

The Purdue TDR apparatus was used to measure the electrical conductivity and dielectric constant. Background on use of TDR in soils can be found in literature[7~10]. A Campbell Scientific TDR 100 tester is connected to a specially designed Multiple Rod Probe (MRP) Head by a 1.8 m long, 50 ohm coaxial cable. The

MRP Head sits on the ring of the mold. The signal generator sends a step voltage pulse to the cable, probe head and probe. Reflections occur as the pulse reaches the top and bottom end of the probe. The apparent dielectric is calculated with the travel time between the two reflections.
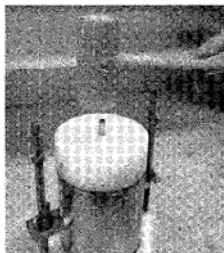
(a) Drive center pin with the aid of a plastic guide

(b) Seat the measurement head for taking TDR measurement

Fig. 1 Sample preparation for TDR monitoring: mold TDR measurement

Analysis of the long-term response of a TDR system allows for calculating the bulk d.c. electrical conductivity, which is related to the long term TDR voltage level[11]. The bulk d.c. electrical conductivity indicates how well the medium can conduct electrical current, and is sensitive to the availability of ions in the medium. Electromagnetic properties (especially the electrical conductivity) are sensitive to temperature. Drnevich et al. [9] provided temperature correction equations for apparent dielectric constant and electrical conductivity.

There are many empirical and theoretical equations relating the volumetric water content of a soil to its dielectric constant. Siddiqui and Drnevich first developed the relationship between the gravimetric water content and the dielectric constant as given by the following equation[12]:

$$\sqrt{K_a}(\rho_w / \rho_d) = a + bw \quad , \qquad (3)$$

where ρ_d is the dry density of the soil, ρ_w is the density of water, a and b are the soil specific calibration constants, which are found to be relatively independent of pore fluid conductivity for situations commonly encountered in field practice[4].

The software PMTDR SM Version 1.4 developed at Purdue University was used to automatically make measurements of apparent dielectric constant and electrical conductivity at fixed intervals of time.

3.2 Measurement of penetration resistance

The strength and modulus or stiffness of lime stabilized soil, are of most interest to engineers. The Dynamic Cone Penetrometer (DCP) is commonly used to estimate strength and the California Bearing Ratio test (CBR) and the Clegg Impact Hammer test are commonly used to find the stiffness of lime stabilized soil. Unconfined compression test on cores from the field or on specimens compacted in the laboratory is frequently used to measure both modulus and strength. Many correlations between the various field tests and unconfined compression test were developed[13].

The stabilized soil is very brittle when compared to the parent soil. The unconfined compression test may not be the best test for such brittle material. The needle penetrometer test ASTM C 403 was used to determined penetration resistance as an indicator of strength. The needle penetration tests were performed at the following time intervals: just after compaction, 12 h after compaction, 24 h after compaction, 7 d after compaction and 28 d after compaction.

4 Results and discussion

4.1 Water content and temperature

From the measurements of water contents after mixing and compaction but before beginning the monitoring, and then again after monitoring water content changes of 0.5% were observed. The water content of a soil reduces, upon addition of LKD due to both hydration and pozzolanic reactions. The hydration of calcium oxide is a very fast chemical reaction and most of it takes place during mixing and compaction. Majority of the water content changes due to addition of lime or LKD can be attributed to hydration reactions. On the contrary, the pozzolanic reactions are slow and consume less water. As the water content of the compacted specimen was measured before and after monitoring, the difference in the measured water contents was probably due to water consumed by pozzolanic reactions because the specimen was sealed with wax.

During the monitoring of test C_2, the room

temperature and sample temperature were monitored. It was found that the temperature of the specimen increased 2℃ after an hour of compaction and then decreased to the room temperature after one day. The temperature changes were not significant and hence no temperature corrections were applied to the measurements of the electrical conductivity and the apparent dielectric constant.

4.2　Electrical conductivity

Fig. 2 shows the variation of electrical conductivity with time for all six samples of Orchard clay tested. The electrical conductivity of the natural Orchard clay with water contents between 15% and 19% are in the range of 50-60 ms/m. Addition of LKD increases the number of ions available for conduction of electricity and hence the electrical conductivity increases significantly. The increased concentration of Ca ions and cation exchange (if any) decrease the thickness of the double layer resulting in flocculation of the clay particles. These two effects (reduction of thickness of double layer and flocculation) caused by the addition of LKD are reflected by a decrease in the electrical conductivity of the sample. From Fig. 2 we see that, the rate of reduction in the electrical conductivity is very high during the initial 20 and decreases thereafter. The bulk electrical conductivity is mainly influenced by the conductivities of pore water and clay particles. Upon addition of LKD, the specific surface areas of the clay particles reduce due to cation exchange and agglomeration. Also, with time the concentration of Ca ions reduces due to the formation of pozzolanic compounds. These changes in the modified soil are associated with a simultaneous reduction in electrical conductivity. From the figure we can also observe that the amount of LKD has a great impact on the changes in electrical conductivity. The lower the amount of LKD in the soil, the earlier the conductivity reaches a more stable value.
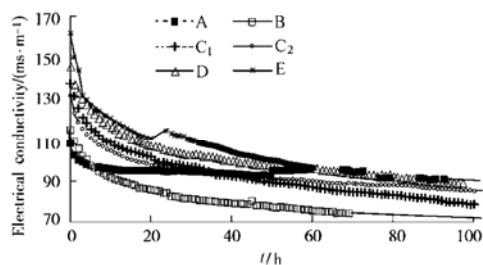

Fig. 2　Electrical conductivity with curing time

We can distinguish the physicochemical reactions from Fig. 2. We postulate that the short-term sharp

decrease of conductivity is caused by the cation exchange and agglomeration, which would be completed after few hours of mixing. The reduced rate of decrease in electrical conductivity in the long term is then caused by the pozzolanic reaction. There is no distinguishable boundary between the two different reactions.

It is possible to fit the curve with the following hyperbolic function for the first 24,

$$EC_b(t) = EC_{b1} - Bt/(A+t)　, \qquad (4)$$

where $EC_b(t)$ is the conductivity at time t after the first measurement, EC_{b1} is the conductivity of the first measurement, B and A are the calibration constants. The conductivity first increases because of the addition of the LKD, then decreases because of the physicochemical processes described above. It does not appear possible to measure the peak value of the initial increase in conductivity. The first measurement should be taken as soon as possible after the compaction, in order that it will be close to the peak conductivity. The calibration constant B denotes the maximum decrease of electrical conductivity. B/A denotes the initial gradient of the decrease of conductivity. The calibration constants of the curve fitting are listed in Table 2. Normalizing Eq. (6) with B and A yields

$$\Delta EC_b(T) = T/(1+T)　, \qquad (5)$$

where $\Delta EC_b(T) = (EC_{b1} - EC_{bt})/B$, $T = t/A$. The normalized curves are plotted in Fig. 3. The hyperbolic function fits all the curves very well.
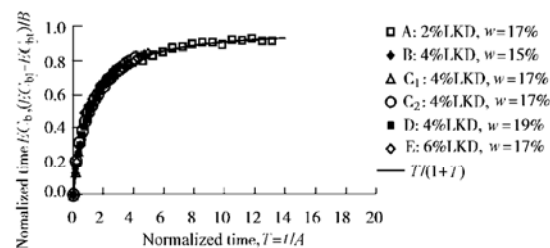

Fig. 3　Normalized electrical conductivity with time

We can define a parameter to show the completion of the cation exchange. Let $\Delta EC_b(T_r) = e^{-1}$, where T_r is the normalized time required for the initial conductivity (EC_{b1}) to decrease to a value of $B\ e^{-1}$ which is the maximum reduction in conductivity during the short-term reactions. The value of T_r for the various tests conducted is provided in Table 2. It is interesting to find that B increases with amount of LKD. These two constants probably are depended on soil type, LKD type,

mixing method, water content, etc. From Table 2 we observe that time durations of the cation exchange are different. According to this approach, 63% of cation exchange takes place during the initial 12, and 80% takes place in 24 after compaction.

Table 2 Calibration constants of curve fitting of electrical conductivity

Test ID	LKD percent /%	Water content /%	B /(ms·m^{-1})	B/A (ms·m·h^{-1})	A /h	$t_r=AT$ /h
A	2	17	14.3	9.37	1.526	2.6
B	4	15	38.3	5.94	6.452	11.1
C_1	4	17	45.9	8.08	5.683	9.8
C_2	4	17	41.5	8.56	4.846	8.3
D	4	19	50.3	7.41	6.784	11.7
E	6	17	61.0	15.11	4.037	6.9

4.3 Dielectric constant

The apparent dielectric constant of all the samples tested is very close to each other. The change in the apparent dielectric constant with time for test C_2 is shown in Fig. 4. The dielectric constant reduces slightly with time. We are currently studying how the K_a measured relates to the free water content with curing.
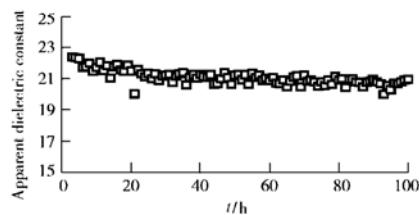

Fig. 4 Dielectric constant with time (Test C_2)

4.4 Needle penetrometer resistance

The penetration resistance of the needle penetrometer is an effective laboratory test to find the strength of the specimen. The results of the tests are plotted in Figs. 5 and 6. There is a sharp increase of resistance immediately after compaction. After 3 after compaction, the increase of the resistance becomes more constant. During monitoring, it was found that the resistance continues to develop after 28 d. The increase of the resistance coincides with the decrease of the electrical conductivity with time. It is encouraging to find that this relationship between the resistance and the electrical conductivity is relatively independent of water content and amount of LKD. The development of the strength is attributed to the physicochemical process in the LKD stabilized soil. The conductivity monitoring clearly shows the physicochemical process. So it is possible to estimate the strength development by measuring the electrical conductivity.

5 Conclusions

The increasing demand for highways and the decreasing availability of satisfactory base and sub-base materials for modern highway and airfield construction have led to the use of cementitious and pozzolanic materials for improving natural soils to suitable construction materials. The by-product materials like lime kiln dust, cement kiln dust are being used for chemical modification of subgrades. Dry density and water content are the only two parameters tested in the field for quality control. The physicochemical processes of LKD stabilized soil were monitored by TDR. The change of electrical conductivity and apparent dielectric constant with time shows the short-term cation exchange and agglomeration, and long-term pozzolanic reactions. The decrease of electrical conductivity coincides with the increase of the penetrometer resistance with time. The amount of LKD present in a modified soil is strongly related to the short term reduction of its electrical conductivity (less than one day). On the other hand, the strength development in the modified soil is related to the reduction in electrical conductivity in long term. Further research will explore the potential applications of TDR technology to the LKD stabilized soil in the field quality control.
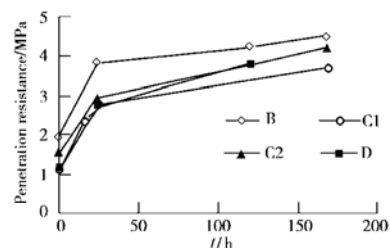

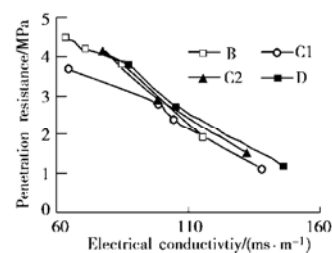
Fig. 5 Penetration resistance with curing time

Fig. 6 Relationship between penetration resistance and electrical conductivity

References:

[1] DIAMOND S, KINTER E B. Mechanism of soil-lime stabilization- an interpretive review[J]. Highway Research Record, National Research Council, 1965, **92**, 83－102.

[2] BHATTACHARJA S, BHATTY J I, TODRES H A. Stabilization of clay soils by portland cement or lime－a critical review of literature[R]. PCA R & D Serial No. 2066, Portland Cement Association, 2003.

[3] BOARDMAN D I, GLENDINNING S, ROGERS C D F, HOLT C C. In situ monitoring of lime-stabilized road subgrade[J]. Transportation Research Record, 2001, **1757**: 3－13.

[4] YU X, DRNEVICH V P. Soil water content and dry density by time domain reflectometry[J]. Journal of Geotechnical and Geoenvironmental Engineering, 2004, **130**(9): 922－934.

[5] GRIM R E. Applied clay mineralogy[M]. New York: Mcgraw-Hill Book Company, 1962.

[6] EADES J L, GRIM R E. A quick test to determine lime requirement for lime stabilization[J]. Highway Research Record, National Research Council, 1966, **139**: 61－72.

[7] BENSON C H, BOSSCHER P J. Time-domain reflectometry (TDR) in Geotechnics: a review, nondestructive and automated testing for soil and rock properties[R]. American Society for Testing and Materials, West Conshohocken, ASTM SPT 1350, 1999.

[8] NOBORIO K. Measurement of soil water content and electric conductivity by time domain reflectometry: a review[J]. Computers and Electronics in Agriculture, 2001, **31**: 213－237.

[9] DRNEVICH V P, YU X, LOVELL J, TISHMACK J K. Temperature effects on dielectric constant determined by time domain reflectometry[C]// TDR 2001: Innovative Applications of TDR Technology, Infrastructure Technology Institute, Northwestern University. Evanston.

[10] YU X, DRNEVICH V P, OLEK J. Predicting strength development of concrete by time domain reflectometry[C]// International Conference on Advances in Concrete through Science and Engineering. RILEM, Evanston, 2004.

[11] GIESE K, TIEMANN R. Determination of the complex permittivity from this sample time domain reflectometry: implications for twin rod probes with and without dielectric coatings[J]. Water Resource Research, 1975, **32**(2): 271－279.

[12] SIDDIQUI S I, DRNEVICH V P. A new method of measuring density and moisture content of soil using the technique of time domain reflectometry FHWA/IN/JTRP-95/9[R]. Joint Transportation Research Program, Indiana Department of Transportation－Purdue University.

[13] JANOO V C, FRICANO A J, BARNA L A, ORCHINO S A. Field testing of stabilized soils[J]. Journal of Cold Regions Engineering, 1999, **31**(1): 37－53.