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Mechanical behavior of chemically treated soils - aging of stabilized granular soils, July 1957

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SOIL STABILIZATION

Report No. 4

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MECHANICAL BEHAVIOR OF CHEMICALLY TREATED SOILS

AGING OF STABILIZED GRANULAR SOILS

BY

Robert L. Schiffman and Ying-Jer Shieh

LEHIGH UNIVERSITY
Fritz Laboratory Report SM-258.4

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A. INTRODUCTION

Since June 1, 1956, Lehigh University has been engaged in a study of the mechanical behavior of chemically treated soil. This investigation is being sponsored by the American Cyanamid Company for the specific purpose of studying the effect of American Cyanamid Stabilizer AM-955 on various mechanical properties of granular soils.

For reasons of scientific convenience, the total study was divided into two broad groupings. The first of these groups was a study of the mechanical behavior of granular soil stabilized with AM-955 in the freshly stabilized state. This study was reported in its entirety previously (1).

The second study, reported herein, is a study of the effects of aging and wetting and drying cycles on the maximum strength of granular soils stabilized with AM-955.

The broad objectives of this study are two-fold. In the first instance, this investigation is intended to determine the effects of aging and wet-dry cycling on strength and to define the conditions, if any, that cause alterations in strength. The second objective of this study is to ascertain quantitative information, in terms of definable concepts of soil behavior, such that, within the scope of this study, this information will be of practical use in the solution of engineering problems.

This study is a pilot study, and as such, is limited in broadness. It is the intent of this investigation to chart future experimental design which will enable an optimum investigation to be carried out.

1. AGING AND CYCLING EFFECTS IN STABILIZED SOIL

Of the three possible actions a stabilizer may have on a soil, AM-955's action is a combination to two types of chemical stabilization⁽²⁾. In the first place, the polymerized gel which fills the voids between particles, place deformation constraints on particle movement. This is the action of an inert void filler. The second type of action that is exhibited is the action of cementation of polymer to the surface of the grain. This action forms a physical and chemical bonding link between grains and introduces force restraints into the grain structural system.

The effect of aging upon strength of the chemically stabilized soil is largely dependent on the alterations in the bonding that produce force restraints. The force system that holds the soil particles together is a three-dimensional network of forces, in which the chemical forms a force matrix within the soil⁽³⁾.

The magnitude of these bonding forces is a function of the chemical properties of the polymer and the physical configuration of the grains with respect to each other. As a first approximation to the behavior in terms of aging, it will be considered that the force restraints on the system will be due completely to the chemical bonds within the polymer. This system will be called the chemical system, while other systems, discussed later, will be referred to as the physical system. The chemical system is schematically represented in Figure 1.

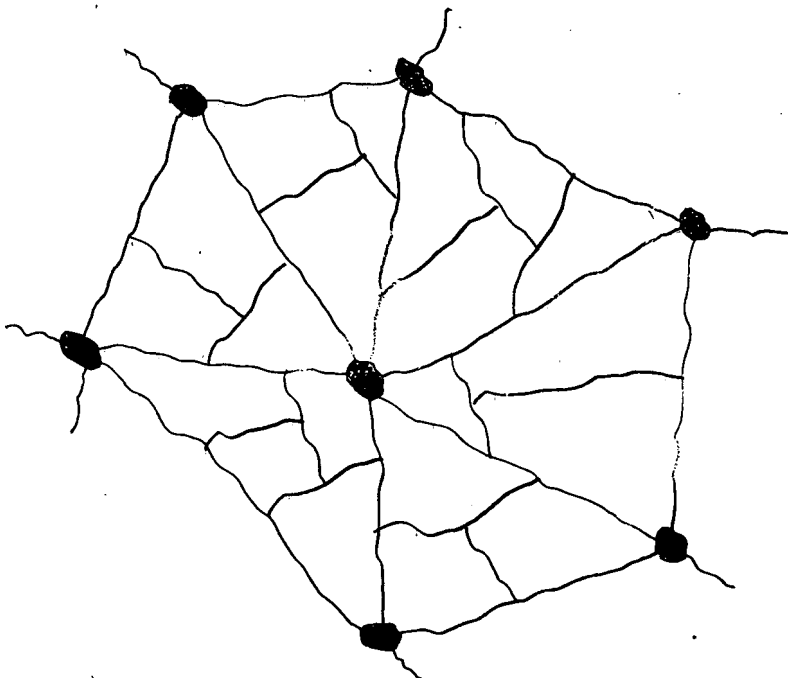


FIGURE 1

SCHEMATIC REPRESENTATION OF CHEMICAL BONDING

The chemical system shown in Figure 1, relies completely for its strength on the chemical bonding of particles, and thus on the strength of the individual bonds, and the configuration of the bond network. The strength of the individual bond is a function of the type of molecule formed and thus the strength of intermolecular bonds. AM-955 in its polymerized state, consists mostly of free water held in a bonded form. The fact that this polymer can form a chemical link between particles must, according to Winterkorn, have its causal roots in the behavior of the water molecule itself. When the free water is removed by evaporation, the nature of the chemical bond is thus altered and thus the strength of the individual bonds change. A similar alteration will occur when water is subsequently replaced into the system. The reasons for this change

and the magnitudes of the change, are beyond the scope of this study and are best studied in the physio-chemical sense.

Certain phenomenological facts are known, however, and gross deductions can be inferred, from these facts. If an AM-955 gel is permitted to dry in the laboratory it shrinks in volume and becomes harder. The shrinking process is uneven and causes cracks in the gel. Upon re-wetting of the gel, there is a softening and an expansion. The cracks, however, do not heal. From this action the gross action of the chemical system can be inferred. With drying, the bond network tends to shorten. The shortening of the bond network is restrained by the configuration of the grains which do not desire to move from their position of stable equilibrium. Thus the net effect of the drying process is to create extensional strains in the bond. The linkage will break if the shrinkage forces are greater than the bond strength. With the re-introduction of water into the system, those bonds which are not broken will be relaxed, but the broken bonds will not heal. A schematic representation of this action is presented in Figure 2.

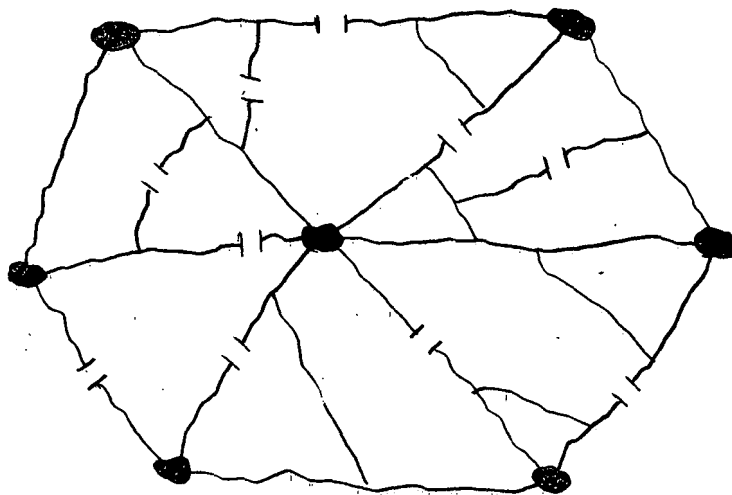


FIGURE 2
SCHEMATIC REPRESENTATION
OF BREAKING OF CHEMICAL BONDS

The configuration of the bond network will affect the behavior only in so far as it alters the geometric force system.

The above discussion presumed that during the drying process, the soil grains remained in a state of equilibrium and thus did not move. The general consideration would be one in which the grains were relatively free to move. In this situation, with drying, there will exist a balance between the shrinking of the bonds and the movement of particles enforced by the chemically induced contraction forces. Thus it is conceivable that the drying process can initiate a volume change in the mass. The stability of particles with respect to volume change alterations, due to changes in the chemical system, will be dependent on several factors. In the first case the uniformity of the stabilization within the soil mass will determine if a given particle is acted upon by a balanced or unbalanced bonding force system. If a balance system is present the bonding forces will be extensional. If, however, the force system is unbalanced the tendency will be to displace the particles. The uniformity will also establish the effects of configuration of bond linkage. The strength of the chemical bonds in a soil-chemical matrix will be a function of their length. The longer the bond, the lower the strength. Thus the distance between particles will affect the strength of the chemical system, with the higher relative densities having higher strengths.

This discussion of a chemical system had as a basic implication, that the chemical bonds within the polymer were equal or of less strength than the chemical-physical bonds between the polymer and the soil grains. Although the relative magnitudes of this hypothesis have not been studied, the fact itself has been.

The polymer without soil binder is in such a weak state that it will visibly deform under the action of its own body forces (gravity). The addition of soil binder establishes forces internal to the soil-chemical mass which counteract the body force effect. The only forces which are additionally present to act counter to the gravity effect are the soil-chemical bonds.

The physical system of forces internal to the soil mass are basically the capillary forces of attraction. For a given particle configuration in the freshly gelled state, and under the assumption that all the voids are filled with chemical, there will be no physical forces on the system other than the initial body forces. As the gel ages under conditions conducive to evaporation, the gel will shrink, and assuming that the particles are not moveable with relation to each other, a capillary meniscus will form between particles as shown in Figure 3.

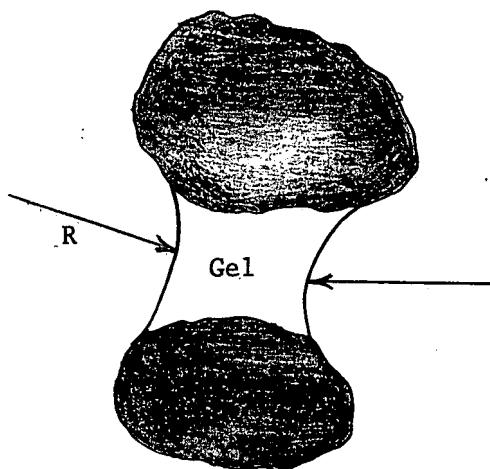


FIGURE 3
EFFECT OF POLYMER DRYING

The physical forces acting on the particles, for a situation pictured in Figure 3, are capillary forces. The magnitude of these forces is inversely proportional to the radius $(R)^{(4)}$. Thus as the gel shrinks, the radius becomes smaller and the physical restraint forces become larger. If the particles are not fixed in space, the shrinkage action may create an unbalanced force system which will translate the particles toward each other, creating a volume change. The magnitude of the physical capillary forces are determinable by the constant of proportionality of the gel or using its accepted term, the surface tension. The surface tension is a chemical phenomenon and depends on the chemical structure of the gel. After a period of drying, if the mass of soil is resaturated, the additional water will be partially absorbed by the gel and the remainder will be in the ungelled structure. The effect will be to reduce the internal capillary forces, but not to dissipate them, since the soil is now in a state where there are three constituents; soil, polymerized gel; and water; and the polymerized gel will continue to exert small capillary forces on the system.

Volume changes due to physical force systems, will occur during drying only when the capillary forces on given particles are unbalanced, resulting in a net force in a given direction. The effect of relative density on the physical forces of drying is such as to alter the magnitude of these forces. At the higher density states the particles will be closer together initially, thus permitting a smaller capillary radius, and higher forces.

It is certain that the chemical and physical force systems are in effect at all but the earliest age of the stabilized soil. The relative magnitudes of these systems will depend on the age of the stabilized soil and the aging history of the soil. This investigation will attempt to

measure these effects, based on the conditions of aging and conditions, whereby the soil mass is alternately dried out and then re-wet.

2. STATISTICAL METHODS OF ANALYSIS

The study undertaken for this report did not involve a rigid statistical analysis. This study is a pilot study and has as one of its purposes the establishment of variants in terms of a future statistically defined study.

Statistical design and analysis of experimental procedures can be broken down into two categories. The first of these categories presumes that there is a definite relation between two or more variables, established by physical laws, and the statistics will be used to define this relationship. In this instance, the problem is statistically undefined and regression procedures are called for. The study of freshly stabilized granular soils used this procedure⁽¹⁾. If the study is to measure the effects of certain controlled factors upon a measurable characteristic of the material in question, then the problem is considered to be statistically defined. This discussion will be limited to a general treatment of statistically defined problems.

If the problem is theoretically defined as one in which the stationary response is desired, methods of sequential analysis can be used. As an example, consider two variants (x) and (y), and it is desired to determine the value of (x) for which (y) is a maximum. In a procedure of sequential analysis, the analysis follows a step-by-step procedure in which the results of prior experiments are used to determine the future experiments. In this example, a few series of experiments of (y) at various values of (x) will determine the gradient of (y) with respect to

(x). Combining this finite difference procedure with an extrapolation method, the subsequent values of (x) can be postulated for the next experiment. Using this procedure, there is no limit to the number of variants that can be considered. The biggest problem in sequential analysis is the uncertainty when there are local stationary points, and only one of many is desired. In these cases an exact knowledge of the theoretical relationship between the variables is necessary to determine the regions of the stationary values desired.

A factorial experiment is one which is designed such that each level of any factor is tested in every combination of levels of the remaining factors being considered (5). Thus in a factorial design it is necessary first to determine what characteristic is being measured, and then the factors involved in this characteristic, and thirdly, the significant levels of the factors involved. This part of the design requires a previous knowledge of the phenomenon being studied and the factors involved. What is unknown is the specific influence of these factors on the phenomenon. The factors involved in the experiment are divided into two classes. The fixed factors are those in which the experimenter selects the levels of investigation prior to testing. In such a situation the generality of the experiment is limited to the levels being investigated. Random factors are those in which the levels of the factor are selected from a random population. In this case, the experimental results can be generalized to a larger set of levels than actually appear in the experimental design. Randomness in this case is specifically defined as the situation in which each level of the generalized set had an equal chance to become one of the levels used in the design.

The above factors are also referred to as controlled variables, in that, their existence and numerical value are determined prior to the actual experiment. If any given experiment were repeated, identically, the probability of identical results is very small. This is due to the experimental error, and the sources of this error are called uncontrolled variables. The design of the experiment to be complete, must include a measure of the effect of the uncontrolled variables. Unless a highly simplified assumption is made of the total response, it will be necessary to make repeated measurements of the total response for each combinations of the controlled variables. This replication of experiments will provide the measure of the effect of the uncontrolled variables. In the design of the experiment, this replication is made in two ways. Within each factor a limited number of replications are made, which is the basic replication. Constancy of certain variables will provide additional measures of replication.

As an example of a factorial design, assume that the mechanical response of a given material (R) is dependent on three attributes (A), (B), and (C). Within each attribute there are 3, 2, and 4 levels of action, respectively. These are the controlled variables. In order to measure the effect of the uncontrolled variables, the experiment will be replicated 3 times. A block diagram of this experiment is presented in Figure 4.

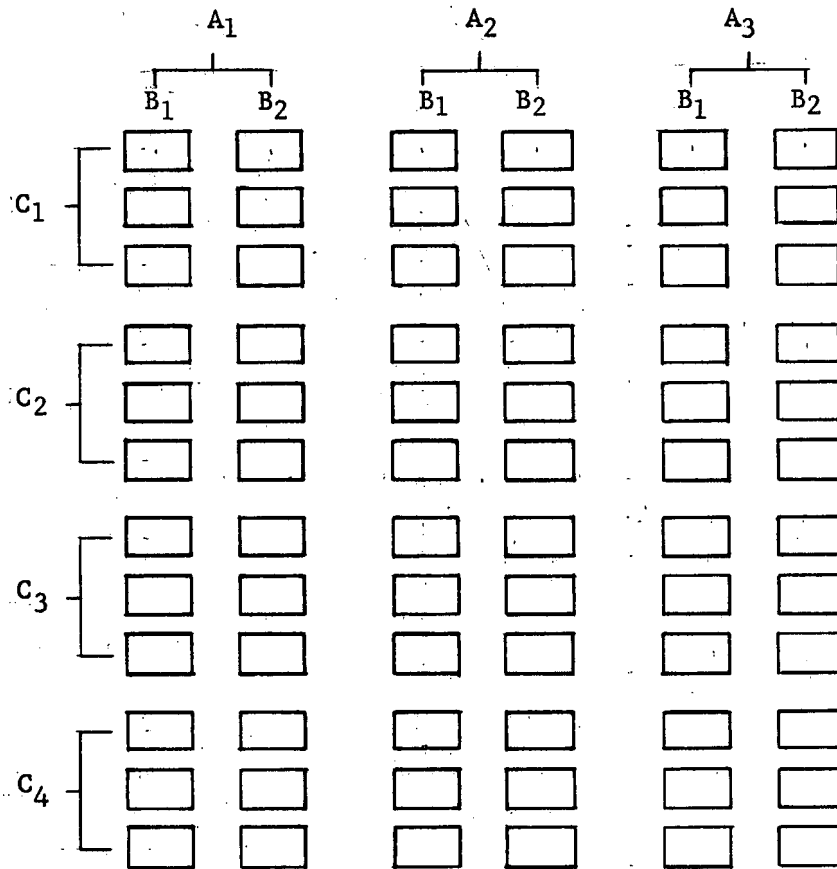


FIGURE 4

FACTORIAL EXPERIMENTAL DESIGN

Each block in Figure 4 represents an individual experiment. Thus, in this example, 72 experiments will be sufficient to analyze the problem. The replication is truly expressed in the stages. The combination of attributes (A) and (C) are repeated on two stages, two (B) values, and three tests from each (B) for a total of 6 replications.

The last specification in the design concerns the manner of the replications. The manner of replication must be made such that each experiment is an independent event drawn from a normal population.

The analysis of an experimental design is based on the concepts of analysis of variance. If tests being made are sampled from several different populations, the techniques of analysis of variance can be used

to detect and estimate fixed relations. Fundamentally, an analysis of variance procedure measures the probabilities of the variations between the individual means of several attributes being drawn from a random population⁽⁶⁾. Thus the statistical tests of an analysis of variance will determine if the various attributes are truly variables, or arise from random chance, and in addition, this analysis will result in quantitative measurements not only of significance of individual variables, but will lead to relations between variables.

The mathematical assumptions inherent in this analysis are as follows:

- (1) The value of each observation is drawn from a random population distributed about a true mean (μ).
- (2) The value of each observation can be considered to be drawn from a total population of variance (σ^2).
- (3) The observation values are independently distributed in a multivariable normal distribution.

The implications of the above assumptions lead to a mathematical model, upon which the analysis is based. This model is generally a linear model which assumes that the measured response is a linear combination of its sources of variation. A typical model based on the example of Figure 4 is as follows:

$$\begin{aligned}
 Y_{ijpq} = & \mu + \alpha_i + A_j + B_r + C_q + (AB)_{jp} + (AC)_{jq} \\
 & + (BC)_{pq} + (ABC)_{jqp} + \epsilon_{ijpq}
 \end{aligned}
 \tag{1}$$

$i = 1, 2 \dots r$

$j = 1, 2 \dots a$

$p = 1, 2 \dots b$

$q = 1, 2 \dots c$

μ = Overall mean effect of all variables upon Y_{ijpq}

Y_{ijpq} = Response of the i th replication of the jpq th factor

α_i = Effect of the i th replicate

A_j = Effect of the j th level of factor a

B_p = Effect of the p th level of factor b

C_q = Effect of the q th level of factor c

$(AB)_{jp}$ = Effect of the interaction of the j th level of factor a, with the p th level of factor b

$(AC)_{jq}$ = Effect of the interaction of the j th level of factor a, with the q th level of factor c.

$(BC)_{pq}$ = Effect of the interaction of the p th level of factor b, with the q th level of factor c

$(ABC)_{j pq}$ = Effect of the interaction between the j th level of factor a, the p th level of factor b, and the q th level of factor c

ϵ_{ijpq} = Effect of the experimental unit in the i th replicate to which the (jpq) th treatment combination has been randomly assigned

The assumptions made on the attributes are that the (ϵ_{ijpq}) terms are normally and independently distributed. Since the analysis is based on deviations from a mean effect, then, in order to construct the model in equation 1 the following must hold.

$$\sum_{i=1}^r \alpha_i = \sum_{j=1}^a A_j = \sum_{p=1}^b B_p = \sum_{q=1}^c C_q = 0 \quad (2a)$$

And, further:

$$\sum_{j=1}^a (AB)_{jp} = \sum_{p=1}^b (AB)_{jp} = 0 \quad (2b)$$

$$\sum_{j=1}^a (AC)_{jq} = \sum_{q=1}^c (AC)_{jq} = 0 \quad (2c)$$

$$\sum_{p=1}^b (BC)_{pq} = \sum_{q=1}^c (BC)_{pq} = 0 \quad (2d)$$

$$\sum_{j=1}^a (ABC)_{jpq} = \sum_{p=1}^b (ABC)_{jpq} = \sum_{q=1}^c (ABC)_{jpq} = 0 \quad (2e)$$

The analysis will test an organized system of hypotheses,

such as:

H_0 : There are no differences among the true effects of the different levels of factor a, i.e., $A_j = 0$; $j = 1 \dots a$.

H_1 : There are no differences among the true effects of the different levels of factor b, i.e., $B_p = 0$; $p = 1 \dots b$.

H_2 : There are no differences among the true effects of the different levels of factor c, i.e., $C_q = 0$; $q = 1 \dots c$.

H_3 : There is no interaction between the two factors a and b,
i.e., $(AB)_{jp} = 0$; $j = 1 \dots a$; $p = 1 \dots b$

H_4 : There is no interaction between the two factors b and
c., i.e., $(BC)_{pq} = 0$; $j = 1 \dots a$; $q = 1 \dots c$

H_5 : There is no interaction between the three factors a, b,
and c, i.e., $(ABC)_{j pq} = 0$; $j = 1 \dots a$; $p = 1 \dots b$;
 $q = 1 \dots c$

The above six statistical hypotheses are tested by making an F-test. Each factor forms an F-value which is the ratio of the mean square of the effect over the mean square of the experimental error. This F-value is compared with an F-distribution, for a predetermined level of confidence. The comparison of these values not only tests the hypotheses (H_0) through (H_5), but a comparison of magnitudes between tests will indicate significance of effects, both qualitatively and quantitatively.

Mathematically, it is a simple procedure to extend the factorial design and analysis of variance to include as many levels and replications of as many attributes as required. The computational procedures necessary for more than three factors, become so large that special techniques become necessary. One very useful procedure is to use a sub-factorial design. A sub-factorial design will break down a parent design of (n) attributes into groups of designs in which no single design of a group has more than three attributes. As an example, consider a design in which there are six attributes; (A), (B), (C), (D), (E), and (F). The mathematical model for this design, including interactions, would contain 63 effects to measure. Instead of handling a single, six-para-

meter model which is highly complex, it would be computationally much easier to work with many two- or three-parameter models, by combining variables.

B. RESEARCH PROGRAM

The research program for this study was designed to study some of the effects of aging of granular soils, when stabilized with American Cyanamid Stabilizer AM-955. The study was partially qualitative, in that, it was designed to uncover the mechanisms at work within the stabilized soil mass, and in particular the mechanics of aging of stabilized soils which are treated with addition polymers. The quantitative portion of the study was designed to give specific information about the effects of aging on the unconfined strength of the soil.

1. EXPERIMENTAL DESIGN

The design of the experimental program was based on a theoretical hypothesis, and statistically it was based on a factorial arrangement of variables.

The theoretical hypothesis of this particular study was specifically as follows:

(1) The change of volume of a given stabilized soil in a given state of relative density as a function of time, is affected by the thermodynamic parameters of temperature, and relative humidity, and the relation of the exposed surface area to the volume of the stabilized soil.

(2) The change of density of a given stabilized soil in a given state of relative density as a function of time, is affected by the thermodynamic parameters of temperature and relative humidity, and the relation of the exposed surface area to the volume of the stabilized soil.

(3) The change of unconfined compressive strength of a given stabilized soil in a given state of relative density as a function of time, is dependent on the rate of drying and the relation between the dry and wet phases of the stabilized mass.

(4) The change of volume of a given stabilized soil in a given relative density state as a function of wetting and drying cycles, is dependent on the number of cycles and the length of time taken for a given phase of each cycle.

(5) The change of density of a given stabilized soil in a given relative density state as a function of wetting and drying cycles, is dependent on the number of cycles and the length of time taken for a given phase of each cycle.

(6) The change of unconfined compression strength of a given stabilized soil in a given state of relative density as a function of wetting and drying cycles, is dependent on the number of cycles and the length of time taken for a given phase of each cycle.

The experiment will test three basic phenomena; the unconfined compression strength, the volume, and the density of the stabilized samples. The response will be measured as a function of the freshly gelled state.

$$\sigma_f(t) = f\left(\frac{\sigma_t}{\sigma_o}\right) \quad (3)$$

$$V_f(t) = f\left(\frac{V_t}{V_o}\right) \quad (4)$$

$$\gamma_f(t) = f\left(\frac{\gamma_t}{\gamma_o}\right) \quad (5)$$

where

$\sigma_f(t)$ = Effect of treatment on unconfined compression strength

σ_t = Unconfined compression strength after specified treatment

σ_o = Unconfined compression strength in freshly gelled state

$V_f(t)$ = Effect of treatment on change in volume

V_t = Volume of laboratory sample after treatment

V_0 = Volume of laboratory sample prior to treatment

$\gamma_f(t)$ = Effect of treatment on density

γ_t = Density of laboratory sample after treatment

γ_0 = Density of laboratory sample in freshly gelled state

Two distinct types of experiments were performed in this study. The first type of experiment measured the effects of drying on the stabilized soil, while the second experiment measured the effects of cycles of wetting and drying.

Statistically, the effects of drying were measured as a regression experiment ⁽¹⁾, in which all experiments were replicated three times over the full range of drying. In this phase of the study, the intention was to determine the trend of behavior. The analysis made was statistically incomplete, in that, an inferred curve was not fitted to the data by an organized statistical procedure. As will be seen, the results of this study are not linear, but exponential, and the time and personnel limitations of the study prevented a complete analysis of the data.

The study of wetting and drying was designed as a factorial experiment, and completed as such. Again, due to the personnel and time limitations of the study, the analysis of variance procedure was not undertaken. The results, however, are in such form that this analysis can be made in the future.

The formulation of both phases of this investigation required the organization of the controlling factors. These factors are listed below in terms of the fixed factors which are set at predetermined levels prior to experimentation, and the uncontrolled variables which require

replication of experiment.

The fixed factors in this experiment and their individual levels of investigation are listed below.

(1) The soil conditions were held to a single level of formulation, in which one soil was investigated at a single relative density state. Thus, in terms of the controlling soil variables, the following were held at one level of investigation.

- (a) Mean grain size held to 0.41 mm
- (b) Range of grain sizes held to 3 sieve sizes
- (c) Relative density of the soil held to the dense state.
- (d) Grain shape held to subrounded
- (e) Shape of grain size distribution curve held to symmetrical or S-shaped

(2) The concentration of the chemical monomer in the stabilized soil was kept at a single level of formulation, in which the amount of monomer was kept at 7% by weight. The catalyst system level was kept at 0.7% of each catalyst.

(3) The gel concentration on a dry weight basis was kept at the single level of formulation of 100% saturation, in the freshly gelled state.

(4) The thermodynamic conditions of drying are held to the single level of formulation, namely, the room temperature at Fritz Laboratory. The average temperature was 75°F, and the average relative humidity was 52%.

(5) The wetting and drying cycle levels set by the drying time.

The samples were dried for a predetermined time and then soaked until the weight gain reached a stable state. The following levels of drying were formulated.

- (a) Thirty days drying - 15 days soaked
- (b) Fifteen days drying - 15 days soaked
- (c) Ten days drying - 10 days soaked
- (d) Five days drying - 5 days soaked
- (e) Three days drying - 3 days soaked
- (f) One day drying - 1 day soaked

(6) The number of wetting and drying cycles was formulated on three levels.

Thus the factorial design of the wet-dry experiment is set. The attributes to be investigated are the drying time which is investigated on seven levels, and the number of cycles which are investigated on 3 levels.

In order to randomize the uncontrolled variables, all tests were replicated three times.

Thus this test is a two-factor experiment which, in terms of strength, will follow the following mathematical model.

$$(\sigma_f)_{ijp} = \mu + \alpha_i + D_j + C_p + (DC)_{jp} + \epsilon_{ijp} \quad (6)$$

$$i = 1, 2, 3$$

$$j = 1, 2, 3, 4, 5, 6, 7$$

$$p = 1, 2, 3$$

where:

$(\sigma_f)_{ijp}$ = Unconfined compression strength ratio of the i th replicate

of the j th drying time and the p th cycle

α_i = Effect of the i th replicate

D_j = Effect of the j th level of drying

C_p = Effect of the p th level of wet-dry cycles

(DC) $_{jp}$ = Effect of the interaction of the j th drying level with the p th cycle level

ϵ_{ijp} = Effect of the experimental unit in the i th replicate to which the (jp) th treatment combination has been randomly assigned

In terms of the complete experiment, the translation of subscripts has been changed for purposes of ease of notation. This translation is indicated in Tables 1 and 2.

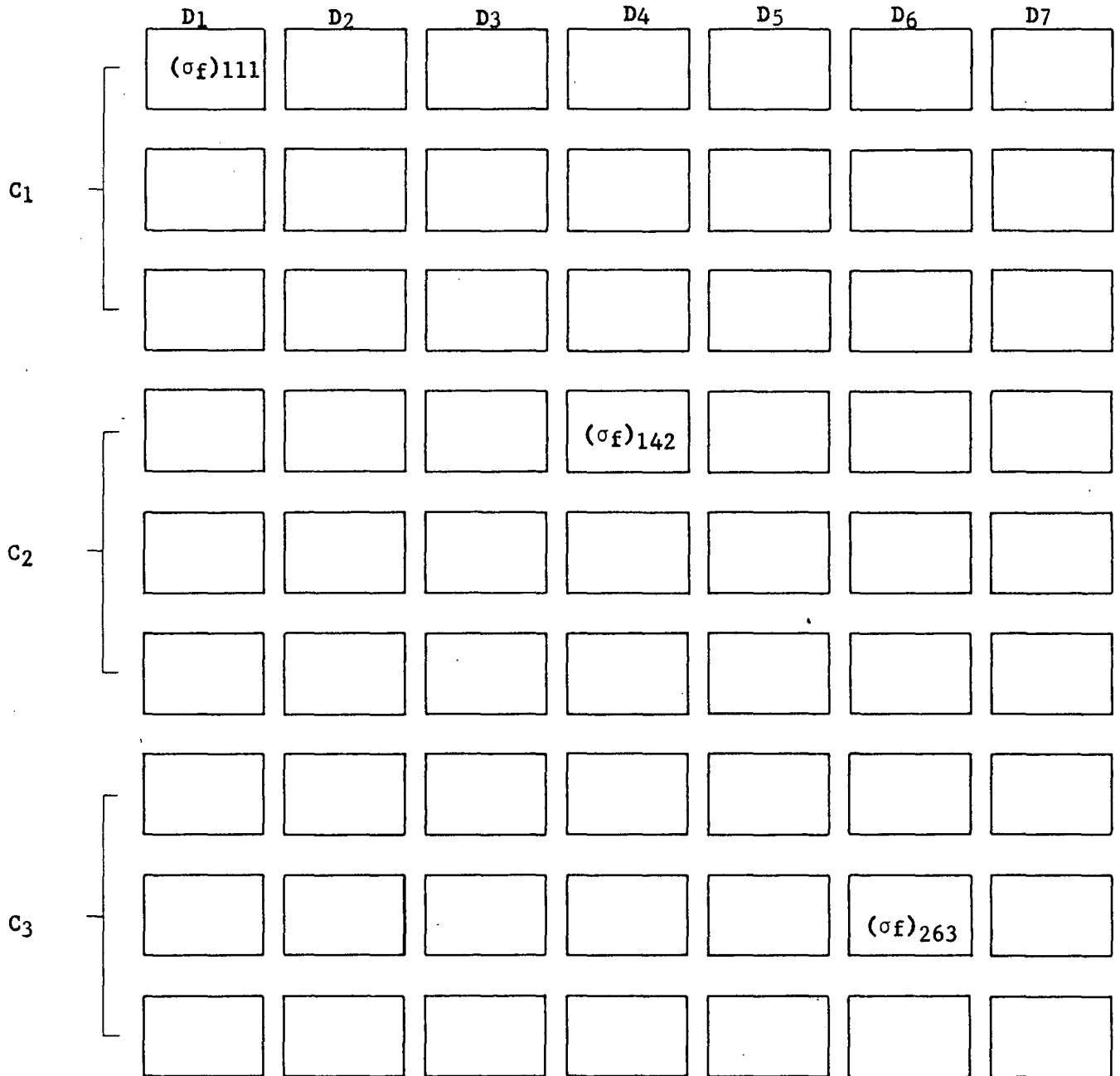
TABLE 1
NOTATION FOR D_j

j	<u>Drying Level</u>
7	30 days dry - 15 days soaked
6	15 days dry - 15 days soaked
5	10 days dry - 10 days soaked
4	5 days dry - 5 days soaked
3	3 days dry - 3 days soaked
2	1 day dry - 1 day soaked
1	0 days dry - 0 days soaked

TABLE 2
NOTATION FOR C_p

p	<u>Number of Cycles</u>
1	1 cycle of dry-wet
2	2 cycles of dry-wet
3	3 cycles of dry-wet

Thus the complete factorial experiment can be represented in Figure 5.



D = Length of Dry-Wet Period
C = Number of Wet-Dry Cycles
 σ_f = Unconfined Compression Strength Response

FIGURE 5

FACTORIAL DESIGN OF WET-DRY
EXPERIMENT

A series of pilot volume density tests were also performed. These tests were performed to ascertain the relation between the exposed area of drying and the volume of the sample on the drying phenomena. The same soil was used as in the other experiments, but due to the difficulties of specimen formation, the soil was formed in the loose state.

For all experiments, a maximum of three mechanical tests were made.

(1) Strain controlled, unconfined compression strength, at a constant strain rate of 0.05-in/min.

(2) Volume measurements with time

(3) Time-dependent weight changes

The experimental program outlined above was carried on in the Soil Mechanics Laboratory of Lehigh University.

2. MATERIAL PROPERTIES

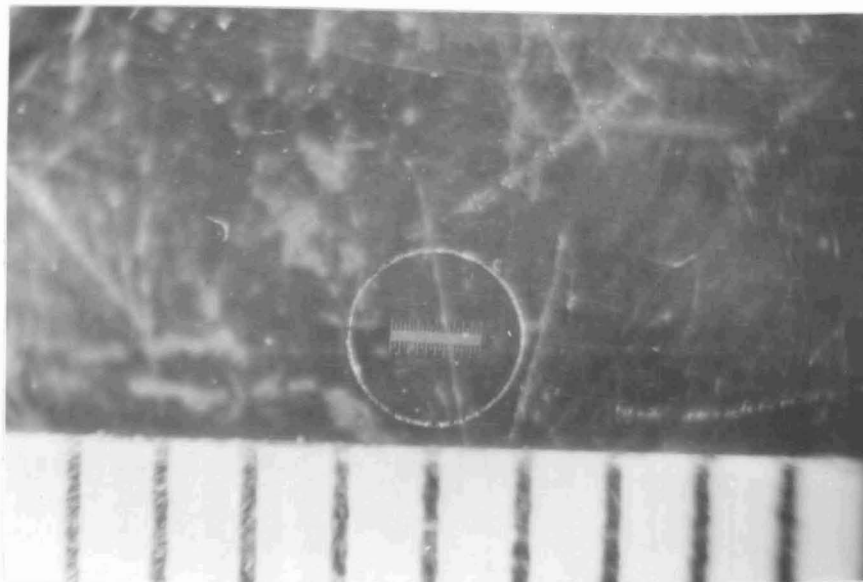
The soil used in this study was a combination of two soils obtained from local suppliers. These soils were river deposits composed mostly of silicates.

The soil used in this study was soil B, used in previous studies⁽¹⁾. Soil B has an S-shaped grain size distribution curve and components as shown in Table 3.

TABLE 3
GRAIN-SIZE DISTRIBUTION OF SOIL B

Sieve No.	Sieve Proportion (%)
16	25
30	50
50	25
100	

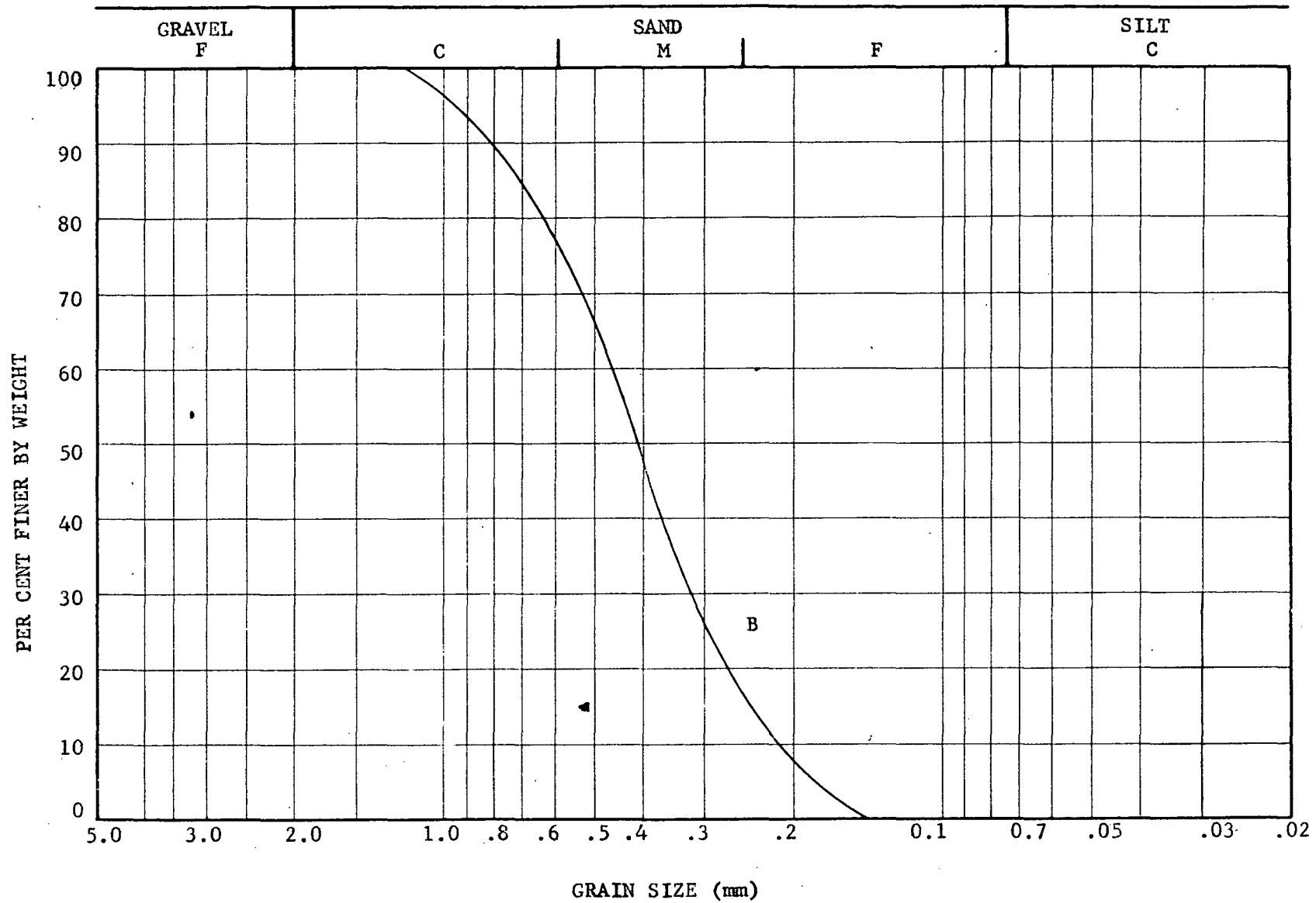
A grain-size distribution curve of this soil is presented in Figure 7. Micrographs of the dry soil are presented in Figure 6.



Scale: 1" = 2.27 mm.

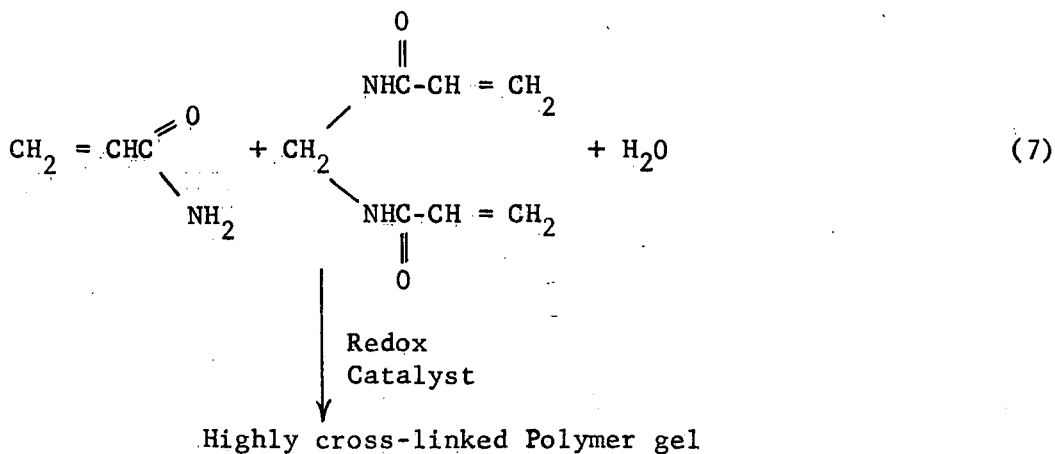
FIGURE 6

MICRO-PHOTOGRAPHS OF SOIL B



GRAIN SIZE (mm)
 FIGURE 7
 GRAIN SIZE DISTRIBUTION EXPERIMENTAL SOIL

The chemical stabilizer used in this study is a product of American Cyanamid Company, and is commercially known as AM-955. AM-955 is a mixture of two acrylonitrile derivatives, these being 95% acrylamide and 5% N,N'-methlenebisacrylamide. This mixture, when dissolved in water in small concentrations, and treated with a redox catalyst, polymerizes to convert the whole solution into a continuous rigid, water-insensitive gel.



Acrylamide is a reactive vinyl monomer of theoretical molecular weight 71.08, and apparent density 1.122 gm/cc. at 30°C.

N,N'-methlenebisacrylamide is a reactive, bifunctional monomer which undergoes reactions which are typical of vinyl or amide groups. Its theoretical molecular weight is 154.17, and its apparent density is 1.235 gm/cc. at 30°C.

The redox catalyst system may be a combination of many chemicals. In this study, the catalyst system consisted of equal quantities of ammonium persulfate as a reducing agent, and sodium thiosulfate as an oxidizing agent.

AM-955 in monomer form, is a colorless liquid which has a specific gravity of 1.005 gm/cc. The viscosity of the monomer is practically identical with water. In this state it was observed to have a small amount of white precipitate.

The gelation time of AM-955 depends on the quantity of catalyst system present. For 0.7% of each component of the system, and distilled water, the gelation time was approximately ten minutes. The specific gravity of the gel was 1.036.

3. TEST PROCEDURE

The procedure used to mix the solution, mold the specimens, trim and handle the molded specimens, as well as the strength testing procedures, were identically the same, as in the study of freshly stabilized granular soil⁽¹⁾.

Pilot tests of various size specimens, were performed on samples initially formed in the loose state. For this series, only volume and weight tests were made. The volume change measurements were performed in two different ways. The first method, in which calipers were used, proved superior to the method of mercury displacement. A photograph of the samples used is shown in Figure 8.

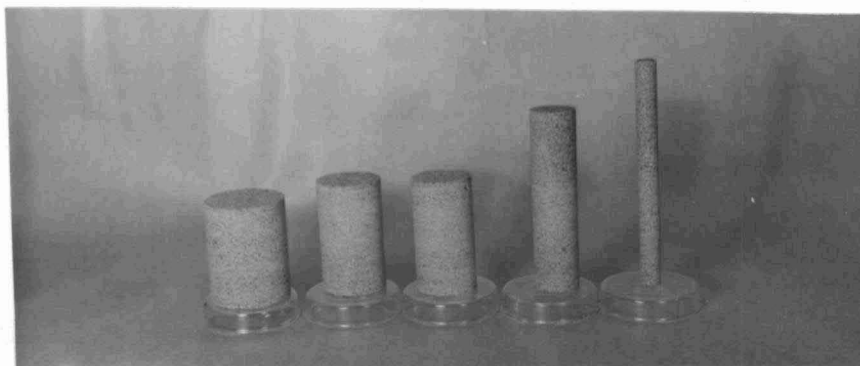


FIGURE 8
PILOT SURFACE AREA SAMPLES

Once the samples for the aging and wet-dry experiment were formed and trimmed, they were set on racks to dry. Each sample was measured daily for weight and volume change. At the end of the specified drying period, the samples were immersed in water to soak. Again, daily weight and volume measurements were made, and when the weight change had stabilized, the cycle was considered to be ended. At the end of the wet-dry cycling each sample was tested to failure in unconfined compression.

C. EXPERIMENTAL RESULTS

The results of the experimental program reported here include the volume and density change with aging, the volume and density changes with alternate wetting and drying, and the unconfined compression strength response with aging and alternate wetting and drying.

1. VOLUME CHANGES

At each stage in the testing program where alterations of the sample were considered, a measurement of volume was made. It was found that the gross volume change of all samples was of such a minor nature as to be considered to be well within the experimental error of measurement, and thus no volume change was measured.

2. DENSITY CHANGES

Measurements of density changes were made on all samples in various stages of aging. These observations were made on samples formed in the loose state in which a variable was the relation between the exposed surface and the volume. The density decay curves of these samples are presented in Figure 9.

Density change observations were recorded during the wetting and drying cycles. Typical density changes for each drying variate are presented in Figures 10a thru 10f.

The change in density for those samples in the dense state which were aged without wet-dry cycles are presented in Figure 11.

3. UNCONFINED STRENGTH RESPONSE

The relationships governing the strength of the stabilized soil was divided into two categories. The first category was the relationship between age and unconfined compression strength. Figure 12 presents some

typical stress-strain curves for the curing process. Figure 13 presents the relationship showing the strength gain with curing.

The second category deals with the strength response of the wetting and drying cycles. In this experiment the response of interest was the relation of unconfined compression strength after treatment to the strength prior to treatment. These relations are presented in tabular factorial form in Figure 14. These experiments were performed in the dense state with relative densities ranging from 84% to 101%. In order to average out the minor effects of relative density, the base initial strength was taken as the average of a series of tests run on samples in the freshly gelled state.

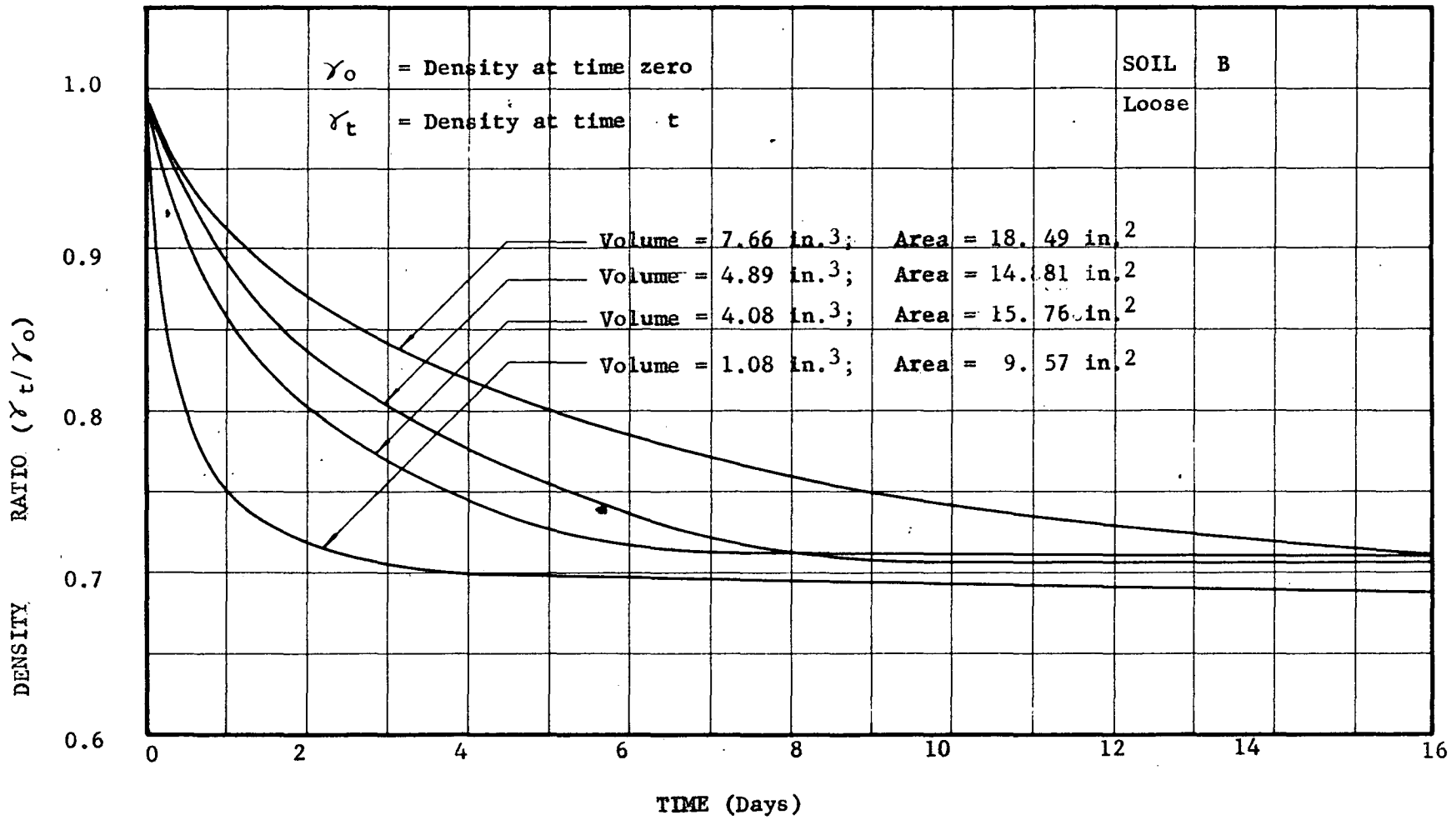


FIGURE 9

DENSITY DECAY AS A FUNCTION OF VOLUME-SURFACE RELATIONS

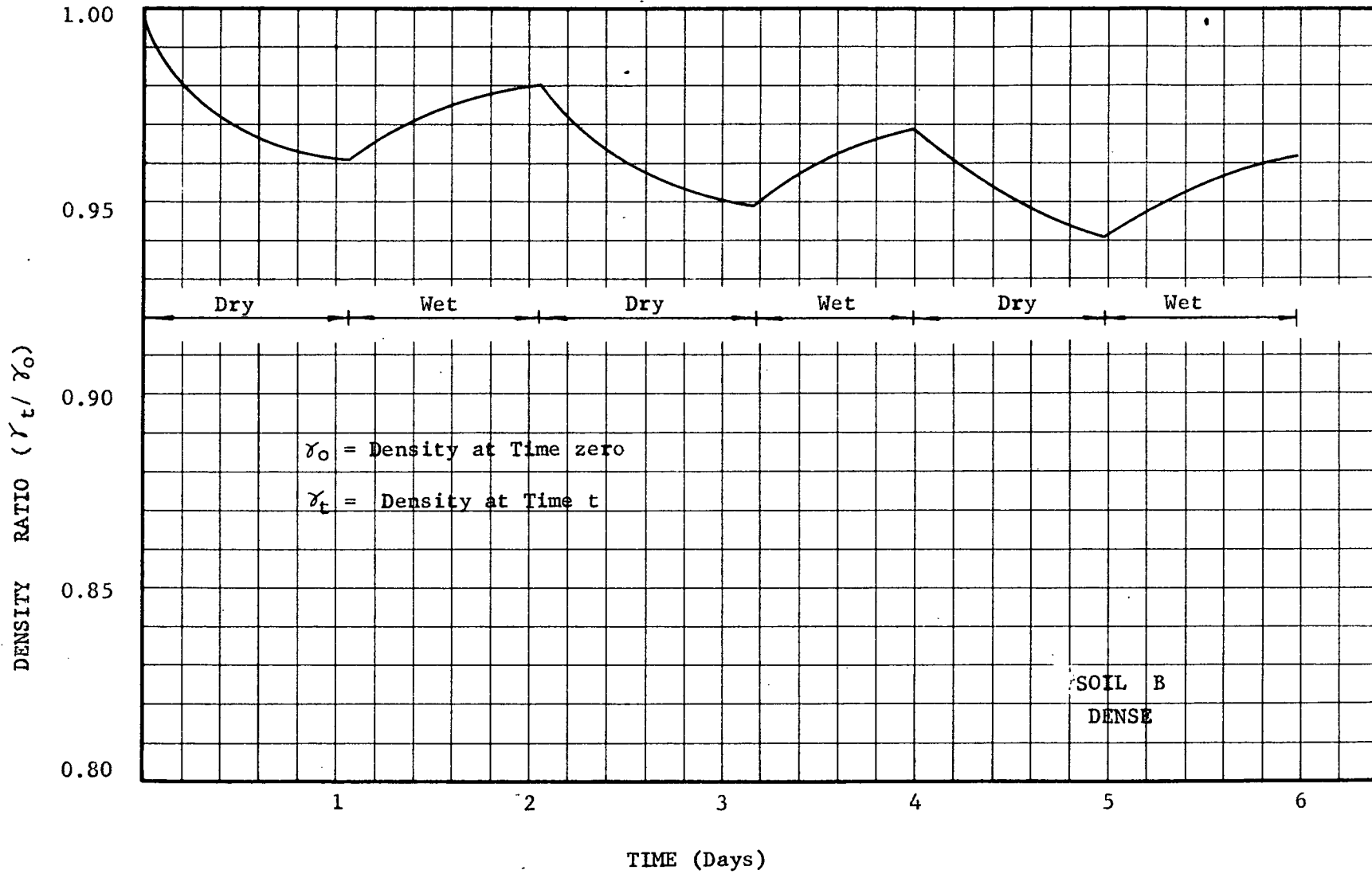


FIGURE 10a

TYPICAL DENSITY CHANGE - 1 DAY - 3 CYCLES

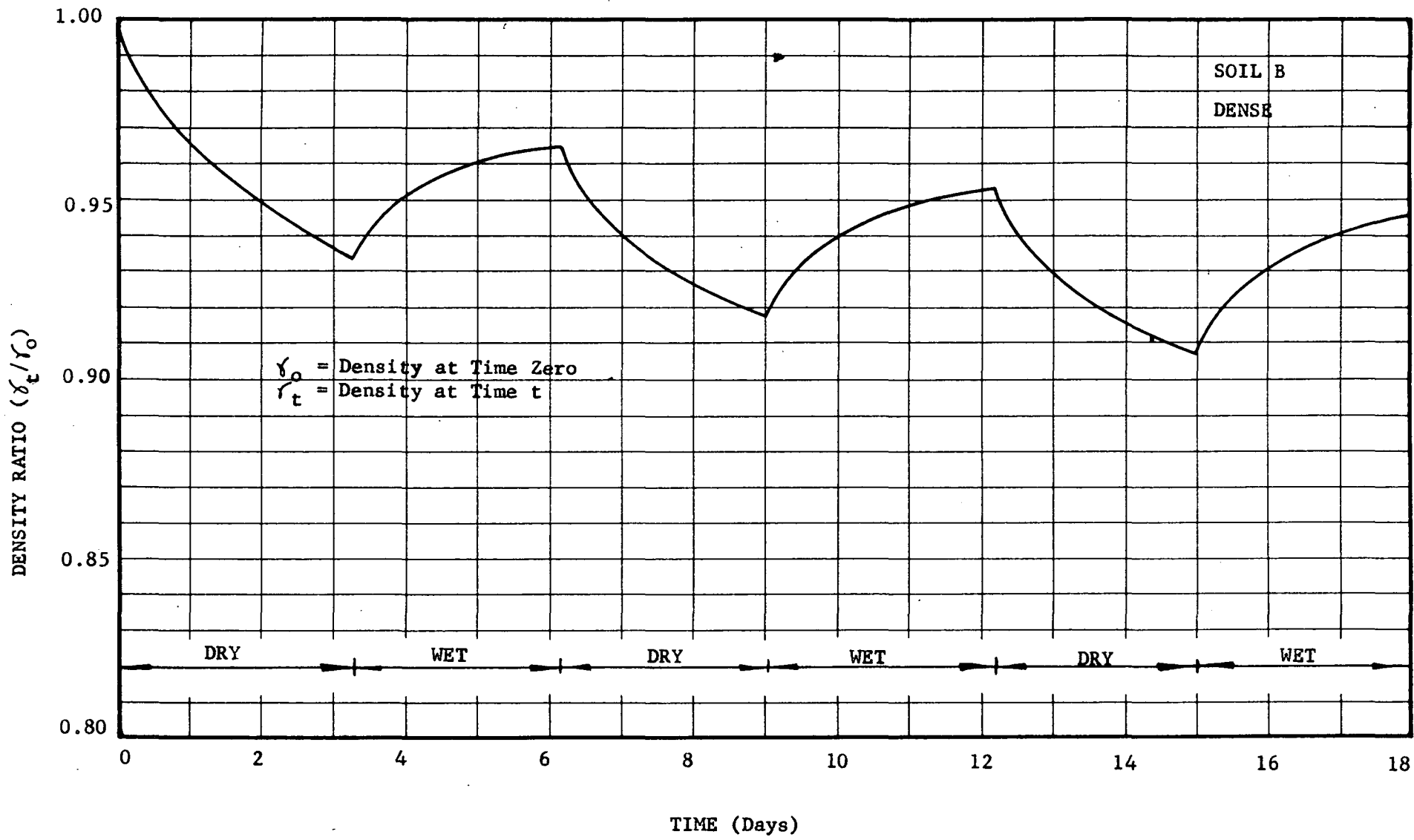


FIGURE 10b
TYPICAL DENSITY CHANGE - 3 DAYS - 3 CYCLES

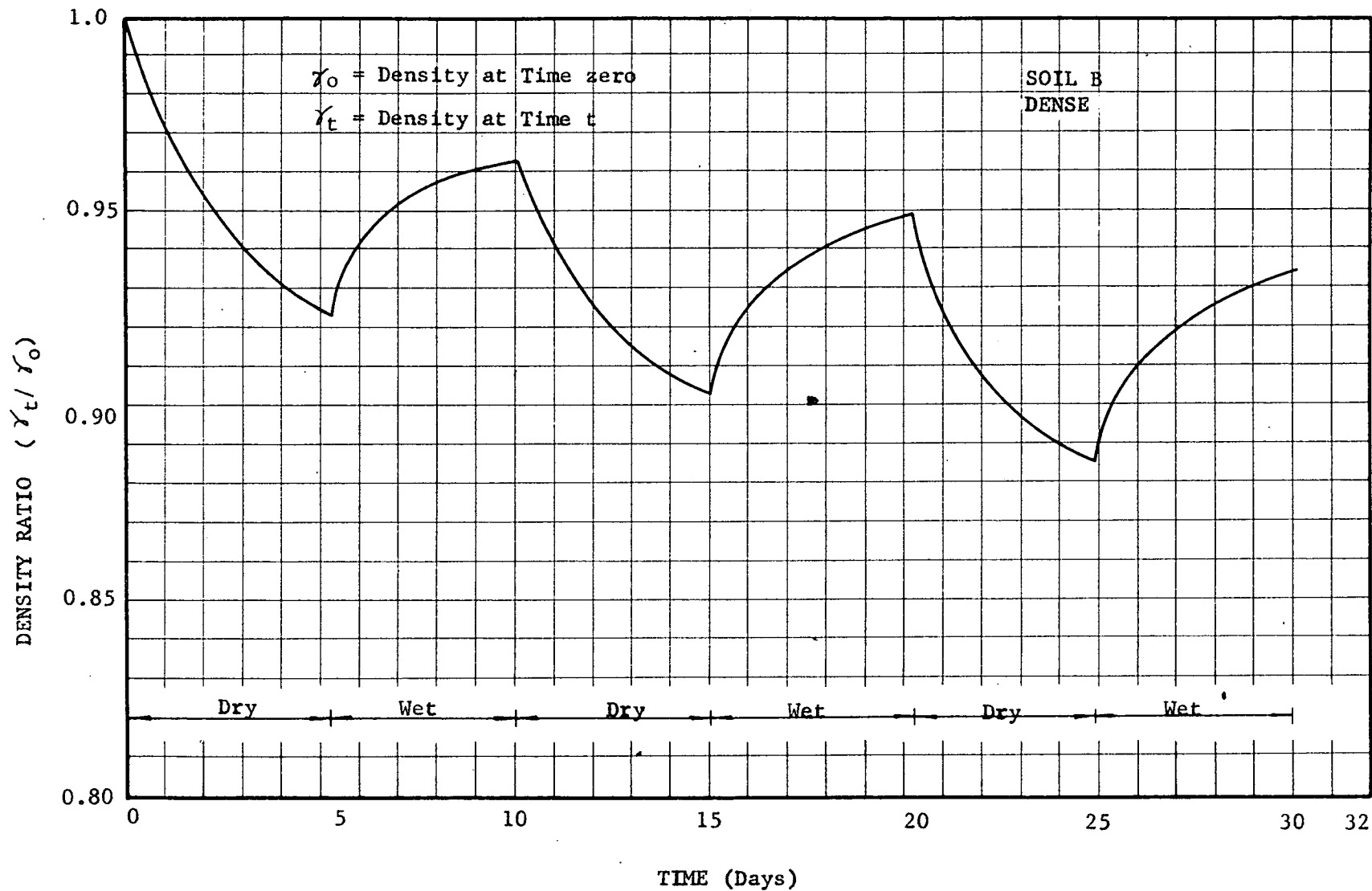


FIGURE 10c
TYPICAL DENSITY CHANGE - 5 DAYS - 3 CYCLES

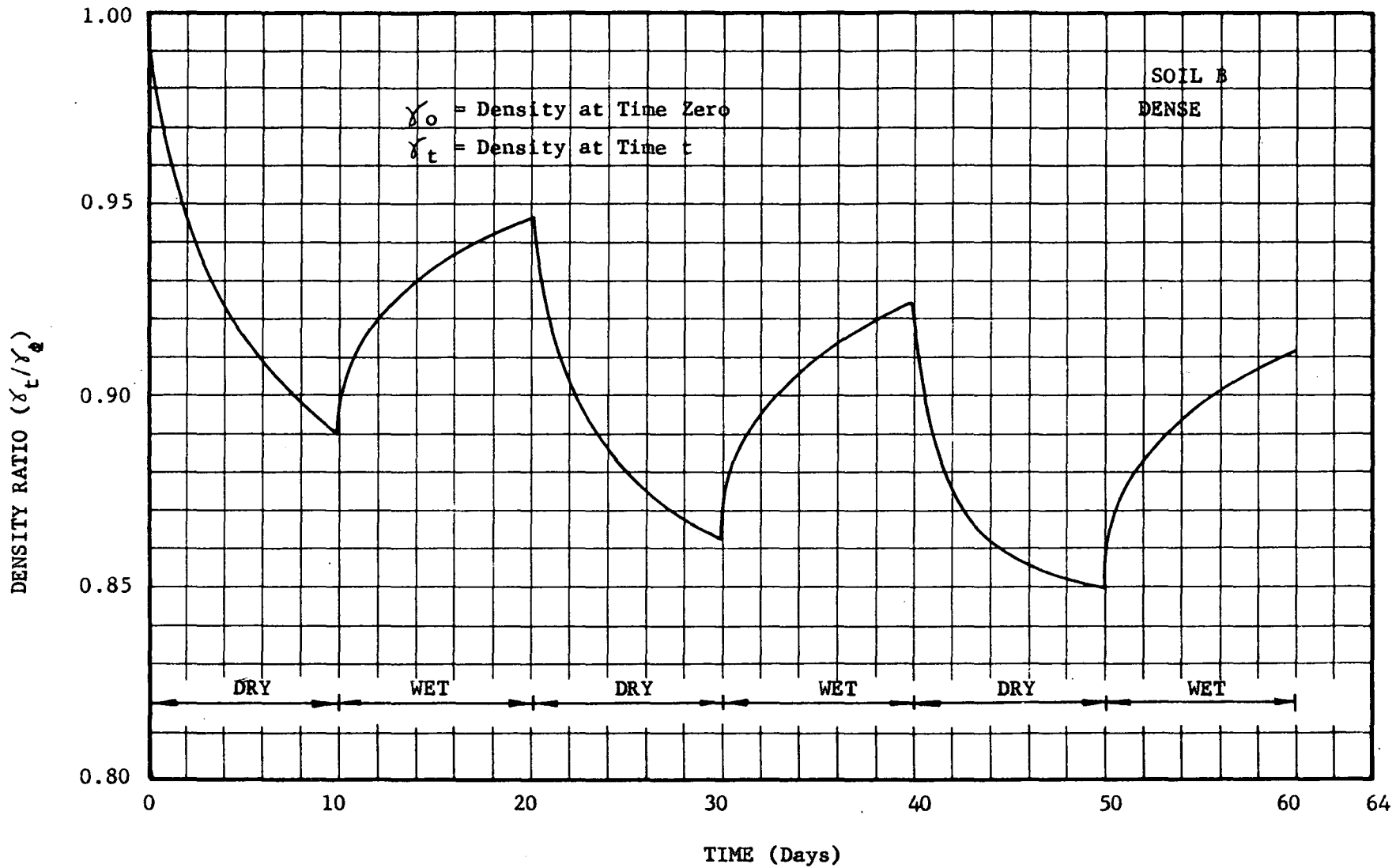


FIGURE 10d
TYPICAL DENSITY CHANGE - 10 DAY - 3 CYCLE

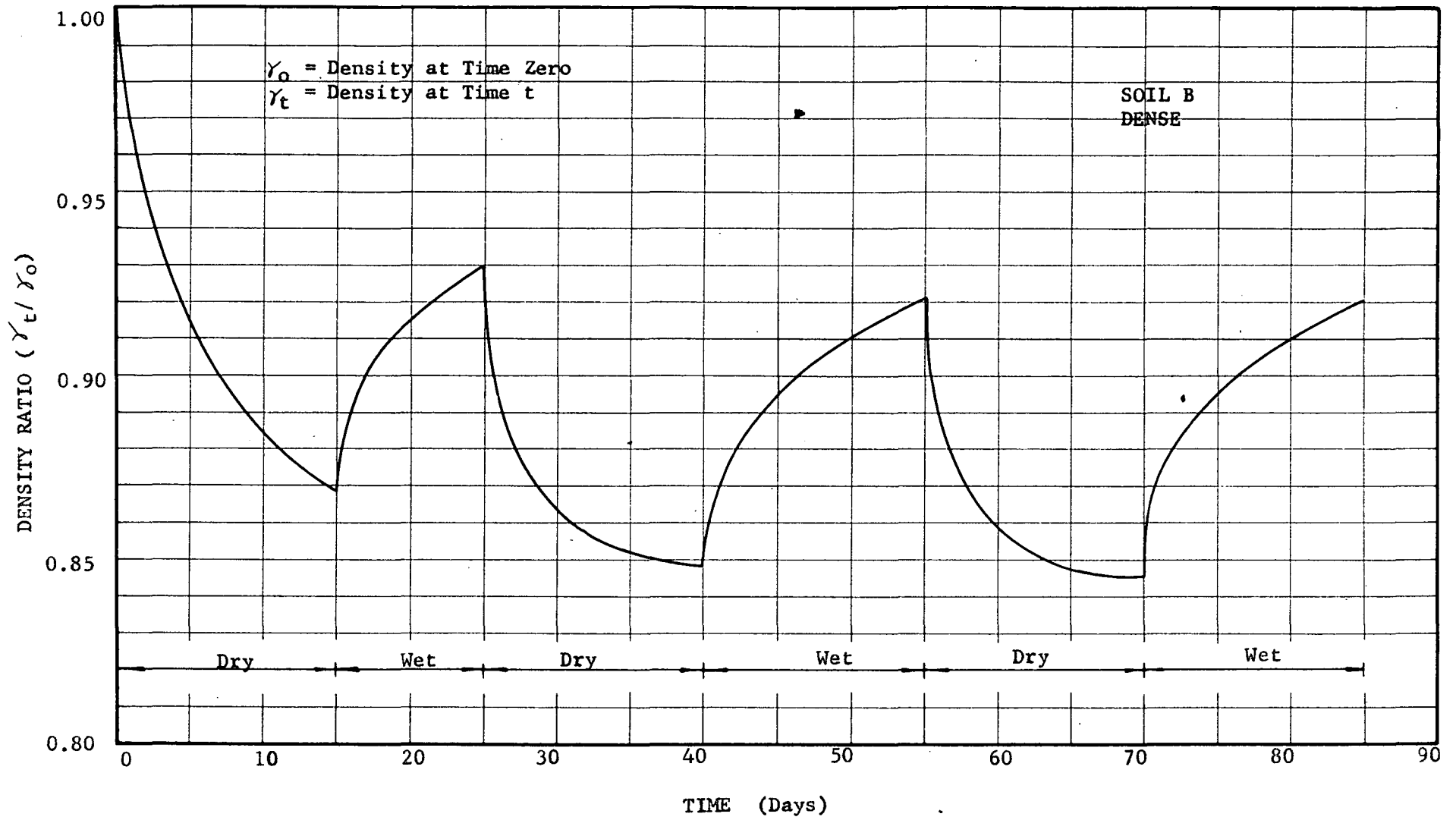


FIGURE 10e

TYPICAL DENSITY CHANGE - 15 DAYS - 3 CYCLES

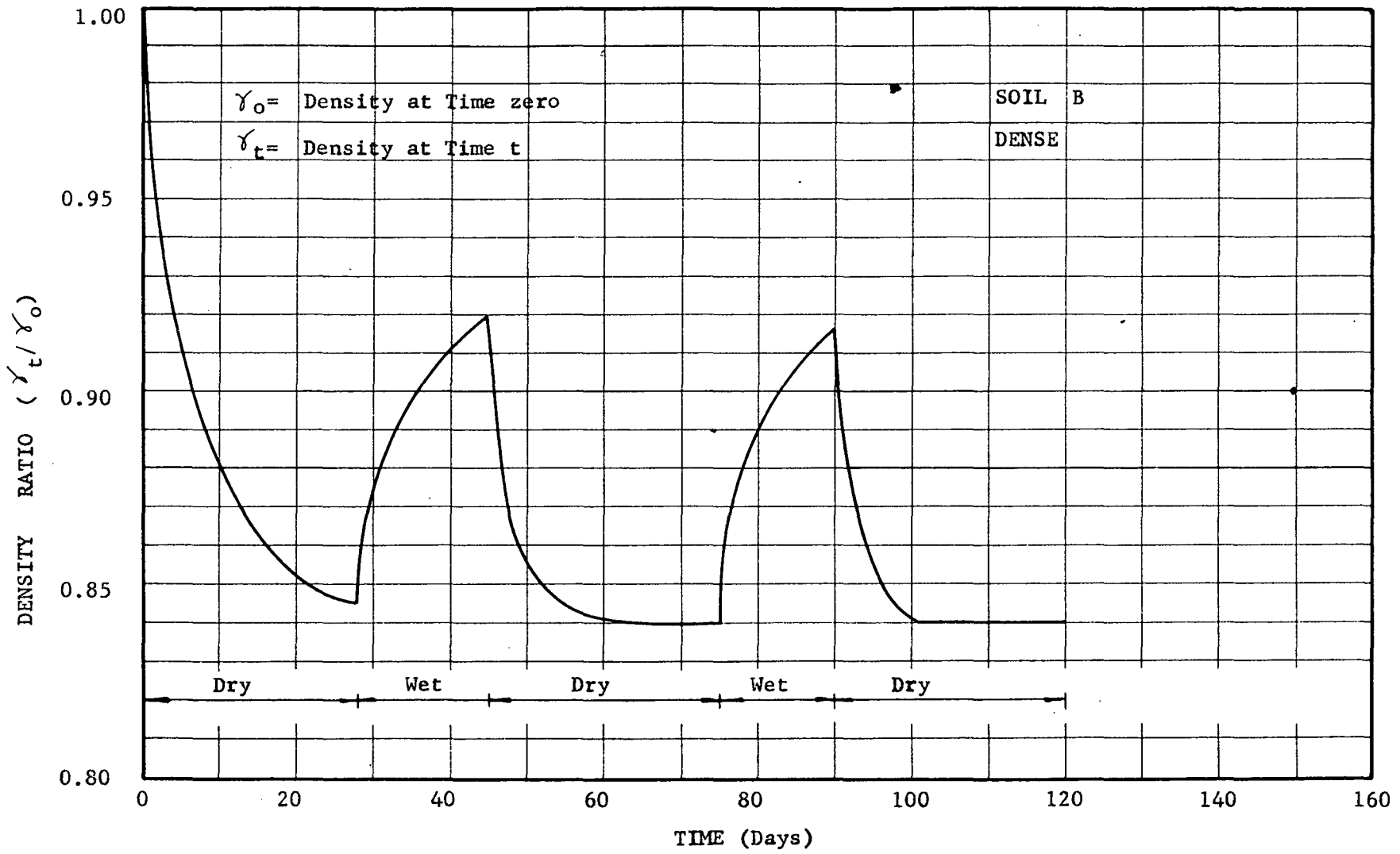


FIGURE 10f

TYPICAL DENSITY CHANGE - 30 DAYS - 3 CYCLES

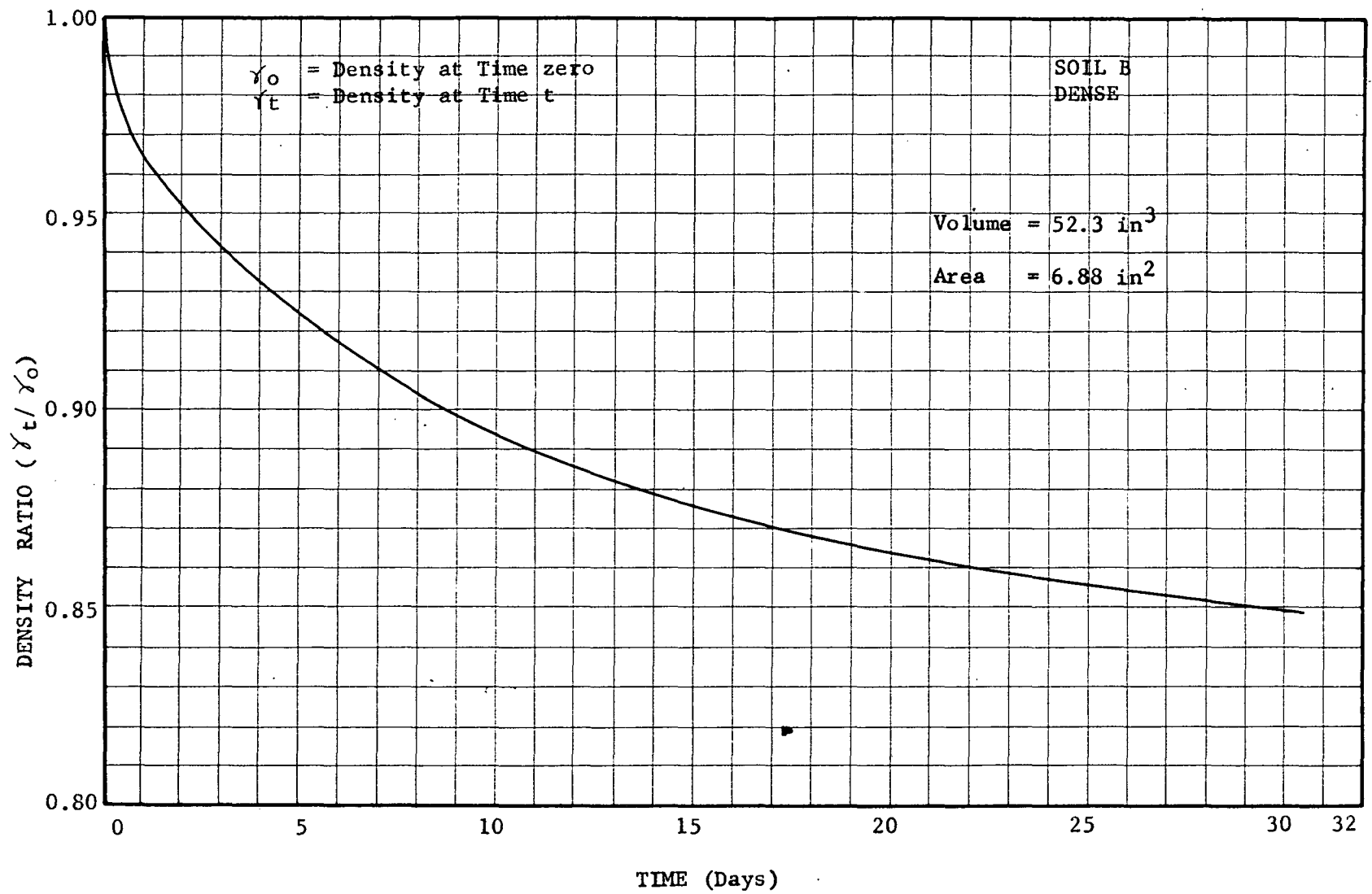


FIGURE 11

TYPICAL DENSITY DECAY

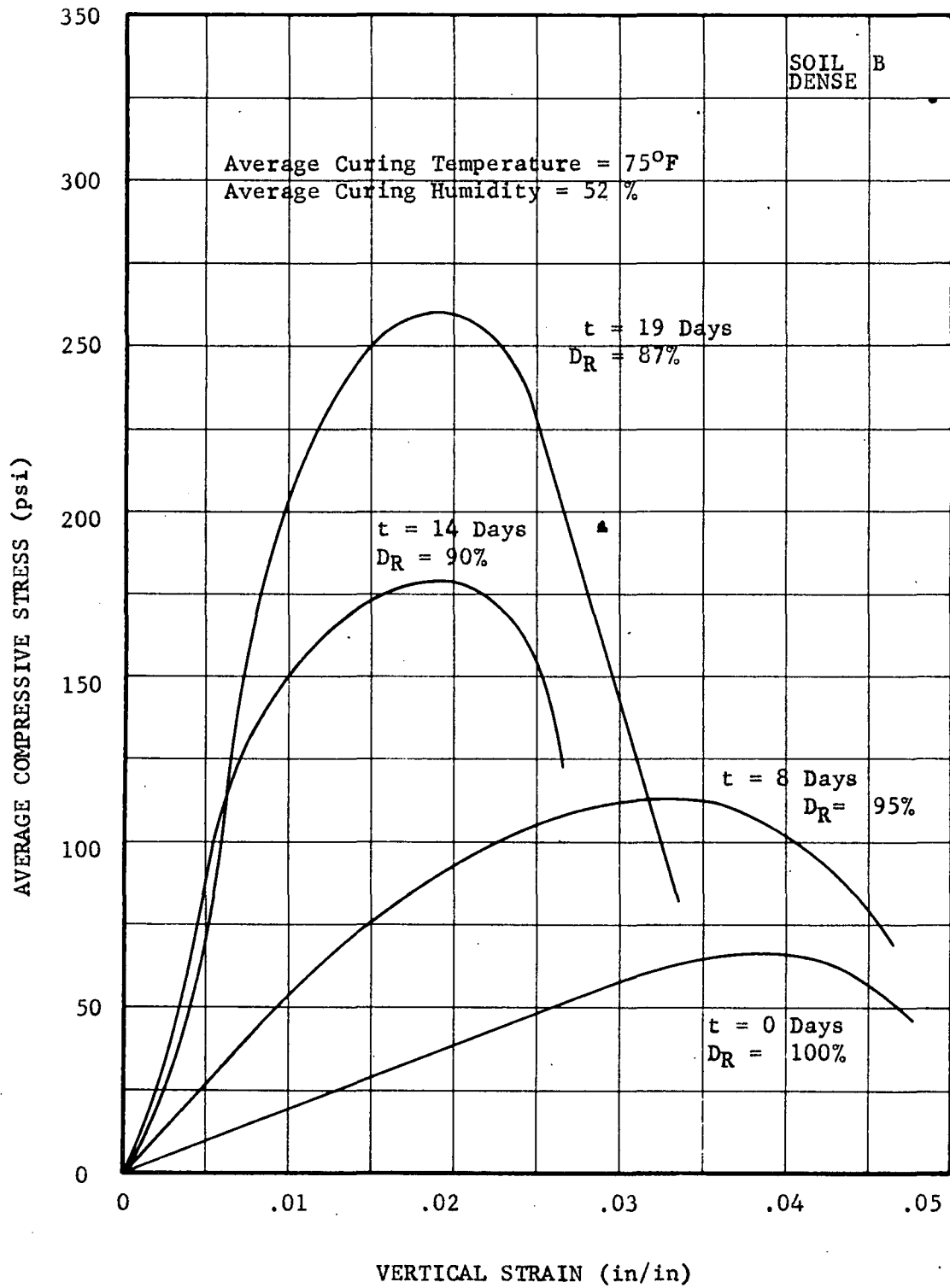


FIGURE 12

UNCONFINED COMPRESSION STRESS-STRAIN
CURVES AT VARIOUS CURING STAGES

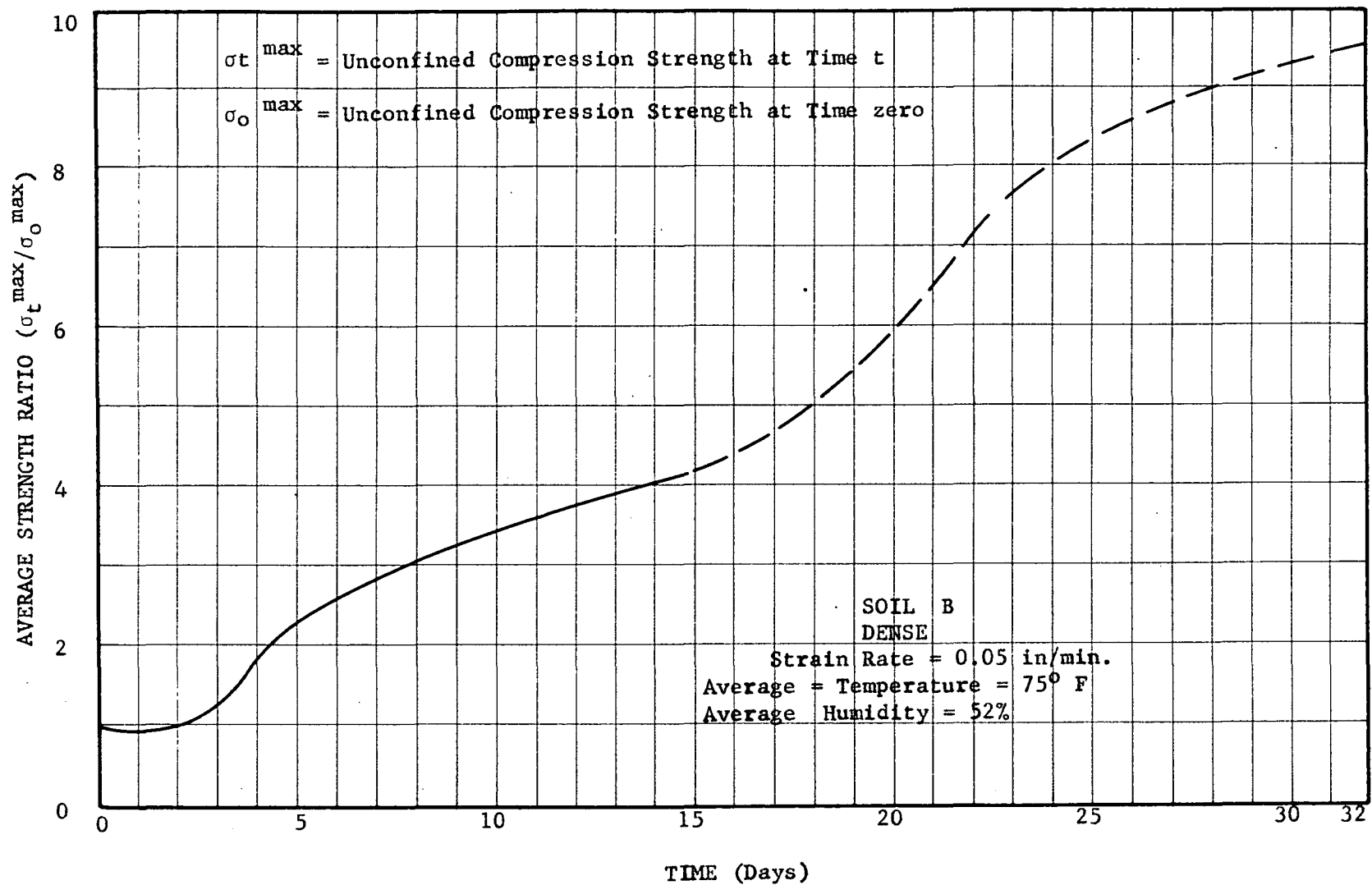


FIGURE 13

EFFECT OF AIR CURING ON UNCONFINED
COMPRESSION STRENGTH

		DRYING PERIOD (Days)						
		0	1	3	5	10	15	30
1	1	1.00	0.92	0.74	0.58	0.59	0.53	0.51
		1.00	0.85	0.74	0.62	0.51	0.50	0.54
		1.00	0.86	0.76	0.63	0.47	0.50	0.52
2	2	1.00	0.79	0.69	0.57	0.56	0.53	0.45
		1.00	0.77	0.67	0.60	0.50	0.56	0.45
		1.00	0.63	0.67	0.60	0.53	0.57	0.50
3	3	1.00	0.77	0.45	0.45	0.57	0.57	
		1.00	0.73	0.58	0.49	0.59	0.56	
		1.00	0.75	0.49	0.45	0.57	0.56	

$\sigma_F = \sigma_t / \sigma_o$
 σ_t = Unconfined Compression Strength after treatment
 σ_o = Unconfined Compression Strength before treatment

FIGURE 14

UNCONFINED COMPRESSION STRENGTH
RESPONSE DUE TO WET-DRY CYCLES

D. INTERPRETATION OF TEST RESULTS

The results of this experimental program can be interpreted in terms of the total response with regard to the variates involved. Although the program of wet-dry experiments was planned so that a detailed statistical analysis could be made, limitations of time and personnel were such that this type of analysis could not be made. Thus the interpretations are limited to those interactions which can be deduced from the test results in a qualitative manner.

1. CHEMICAL STABILITY AND GEL TIME

Certain stability phenomena was observed in the course of this investigation. Since the results reported in previous sections are in the form of ratios these phenomena are not apparent, nor are they applicable to the reported results of this report. In the early stages of this investigation two facts became known. The first of these was that the absolute strength of the freshly stabilized soil at the percentage being used was less than the strengths ascertained in previous work⁽¹⁾. This characteristic was studied intensively in a corollary investigation on the relation between strength and percent of monomer. It was found that for percentages of AM-955 of 10 or greater the unconfined compression strengths were linear relations with the chemical percentage. This linearity also extended to the results attained in Report No. 3⁽¹⁾, and confirmed the results of Karol⁽⁷⁾. It was concluded after a careful series of experiments in which many variables were eliminated that the source of this apparent difference in experimental results lay in the stability of the chemical which had been in the laboratory a period of more than nine months. Since higher percentages of AM-955 at an advanced age resulted in strengths consistent with the previously recorded lower percentages, it was concluded that

the effects were within the catalyst system. There is abundant information to show that the strength is not related to the catalyst system, and thus only one remaining interpretation is possible. It was reported by Karol⁽⁷⁾ that with concentrations greater than 4% the gel properties will have consistent reproduceable properties. If the catalyst system deteriorates with age it is likely to affect, chemically, the polymerization system, and quite possibly the reported percentage may have to be increased, or modified, as a function of age of catalyst. It should be emphasized that the above interpretation is tentative and is subject to experimental justification.

In this particular investigation one departure was made from the previous investigation⁽¹⁾. In the study of freshly stabilized soils the vast bulk of the soil used was obtained prior to the experimental program. In this investigation the soil was obtained from a single local supplier, in small increments during the investigation. Sufficient checks were made of each new batch of soil to assure that there were no strength differentials occurring from the soil. The time of gellation did fluctuate with soil differences, with times as fast as two minutes and as slow as thirty minutes. The time of polymerization was noticed to be slower for the coarser soil. On this basis it is tentatively believed that the constituency of the finer fractions of a soil, and in particular the small amounts of chemically active minerals, will have a profound effect on the reaction time of the chemical system.

2. EFFECTS OF AGING

The first apparent factor about curing of granular soils that are stabilized with AM-955, is the fact that these soils do not undergo any change in volume either in the loose or the dense state. The lack

of volume change with aging is contrary to results obtained by other investigators⁽⁸⁾. Other investigations imply that while there may not be any volume change in the dense state, the drying of the gel will certainly impose a change of volume in the loose state due to the internal capillary forces. This condition will be true if these forces, when summed over a basic surface, are unbalanced. If, however, the forces are balanced with a zero resultant, then it does not necessarily follow that a change in volume must exist. A change in volume must be associated with a particle movement, and in order to move soil grains there must be an unbalanced force system on the grain structure. The results reported by the Cyanamid group in which volume changes were noticed, were, to the best of the authors knowledge, based on experiments in which soil was randomly poured en mass into the ungelled chemical. This method of sample preparation leads to partial saturation in which random air pockets of varying size are internal to the sample. These air pockets will inevitably lead an unbalanced force system and thus a volume change. The samples in this study were prepared very carefully and as such the saturation was between 94 and 100 percent. Thus in drying there was an average over all net system of balanced forces and thus no subsequent volume change.

The net major effect of drying is basically not to alter the volume, but is one in which the unit weight is altered to a point in ultimate drying where it approaches the dry density of the soil. As the gel ages in a thermodynamic system conducive to drying, the chemical bonds in the polymer will break at a point forming a macroscopic crack in the gel. The bond break will be extensional and due to the stretching of these bonds as free water is driven from the system. Once a

crack is formed a new evaporation surface is created and the crack will propagate much like a tension crack in metals⁽⁹⁾. This crack will propagate internal to the gel system, since the surface tension, namely the chemical-physical bonds between the chemical and the soil particles, will hold the gel to the particle. The end result will be a system of air voids in the soil and a capillary force system holding the soil grains. The drying phenomena is schematically shown in Figure 15.

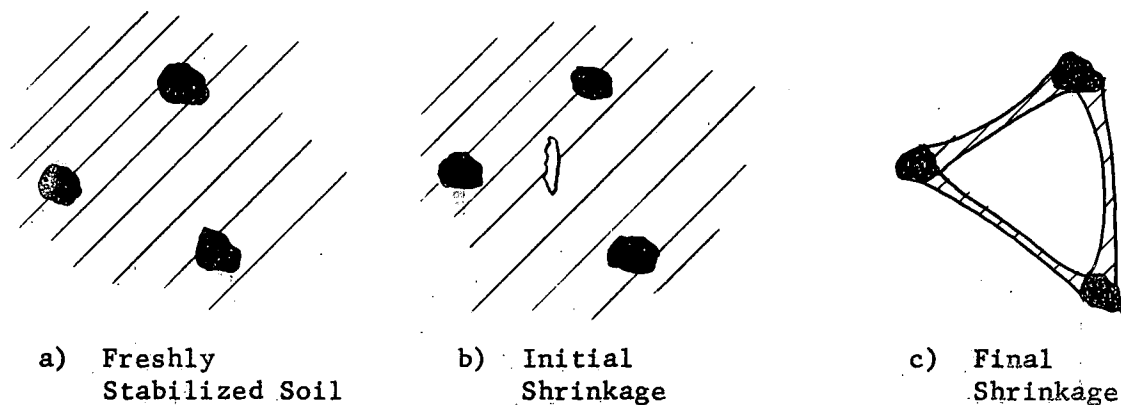
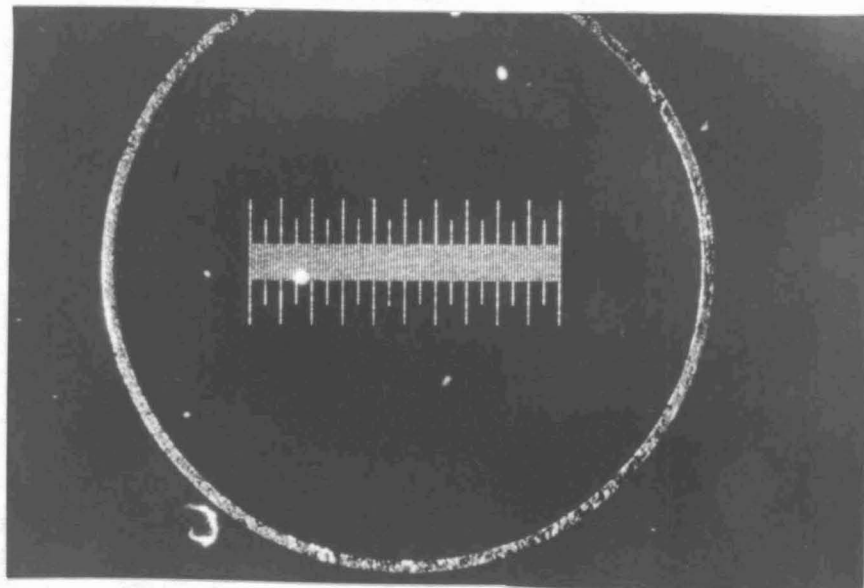
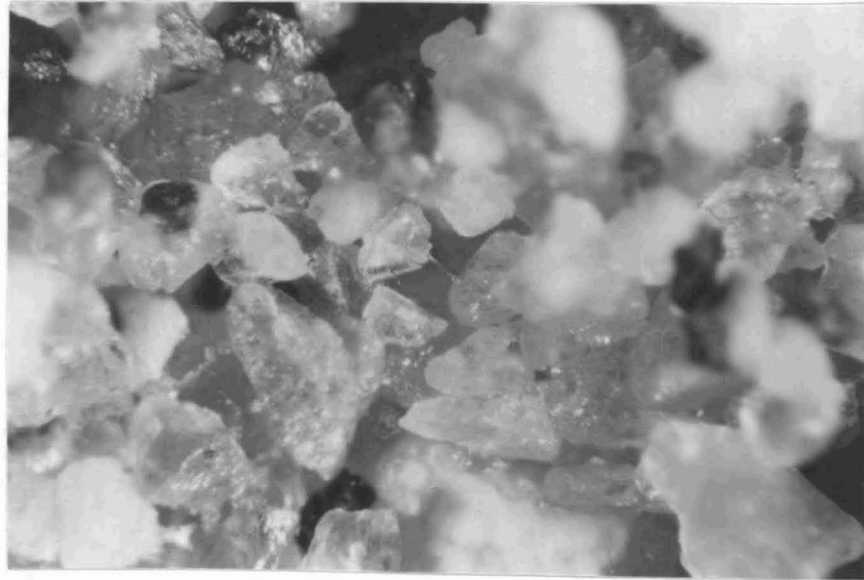


FIGURE 15

SHRINKAGE EFFECTS

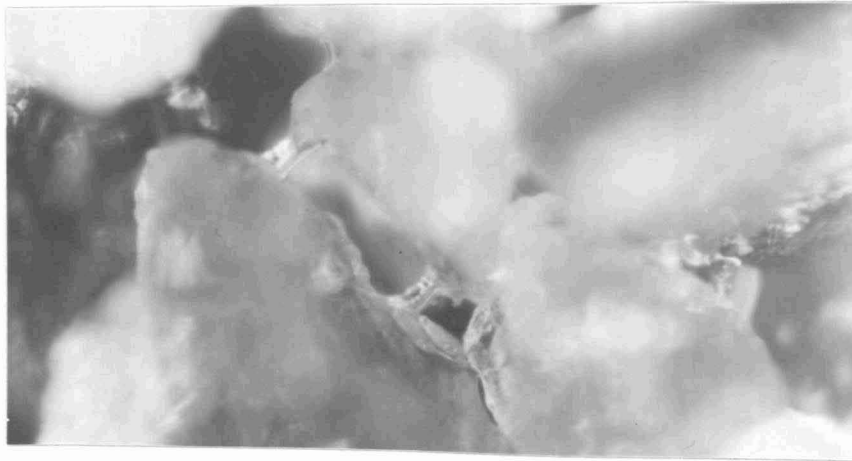
Micro-photographs of the shrinkage phenomena are presented in Figures 16 and 17.



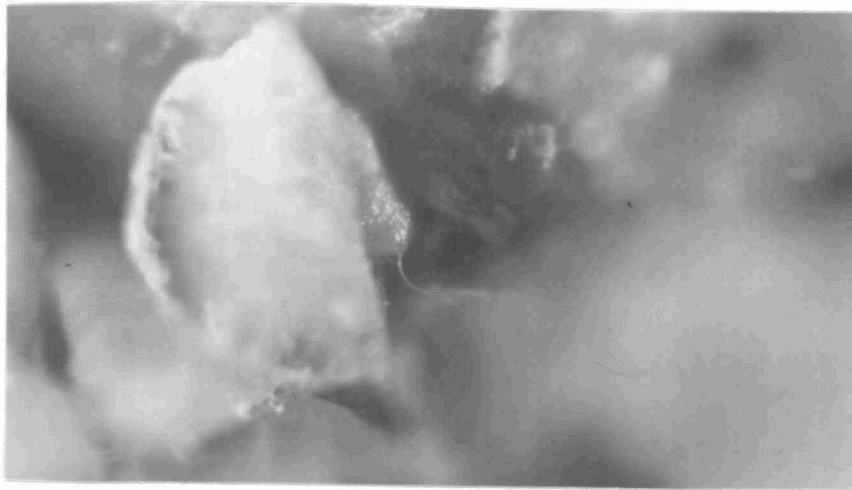
Scale: 1" = 0.625 mm.

FIGURE 16

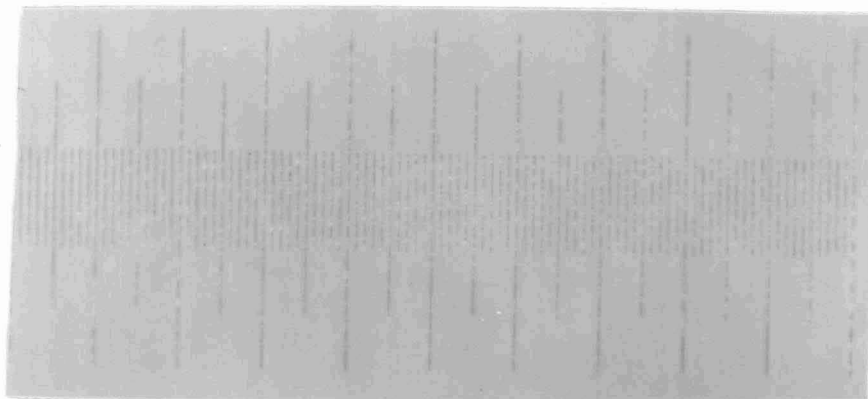
MICROPHOTOGRAPH OF STABILIZED SOIL
IN FINAL STATE OF SHRINKAGE



Early Shrinkage



Final Shrinkage



Scale: 1" = 0.229 mm.

FIGURE 17

MICROPHOTOGRAPH OF STABILIZED SOIL
DURING SHRINKAGE

The strength of the stabilized soil, as shown in Figure 13, will be greatly enhanced by the capillary forces that are set up and will be of the order of ten times the freshly stabilized strength.

A comparison of Figures 9 and 11 with Figure 13, show that as the density decreases, the strength will increase. This is due to the loss of free water, creating air voids and a capillary force system. The change in density itself is thus the macroscopically controlling mechanism for strength increase. The density fluctuation is a thermodynamic phenomenon which is controlled by the amount of surface exposed to evaporation pressure. As would be expected, the drying proceeds inward starting at the boundary surface. The drying process can be broken down into two separate and distinct systems. The first system is that of elemental drying in which a small unit of stabilized soil dries, forms air voids and creates new surfaces of evaporation in which a thermodynamic unbalanced moisture vapor pressure exists. The mass drying phenomena is the process by which, over a finite period of time, whole volumes are of a relatively constant density but differ from adjacent finite volumes. In laboratory cylinders the drying naturally follows a cylindrical law in which the drying is inward in concentric rings. After any given period of time the dry outer ring may be peeled away from the inner wet portion. This difference between elemental and mass drying is probably due to the difference in the rates of drying in these two masses. As indicated by Figure 9, the density change is dependent on the air-exposed surface. As the voids in the outer-ring increase, the rate of drying will increase. Since drying works inward, the outer mass will always be drying at a faster rate than the inner portions. Thus, by the time the air voids have penetrated the outer mass, to initiate a drying process, the outer portion will have reached

a state of equilibrium. A photograph of the concentric drying phenomena is shown in Figure 18.

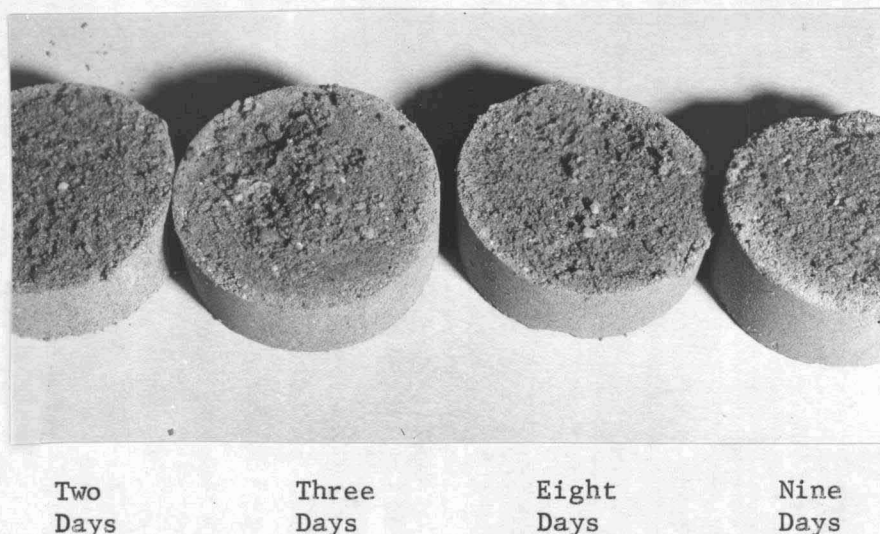


FIGURE 18

CONCENTRIC DRYING PHENOMENA

The rate of increase of unconfined compressive strength as shown in Figure 13, is a function of the strain mechanism and the rate of drying. The material during any phase of drying is a two-phase material consisting of a hard outer shell and a soft core. These two materials are joined in such a manner as to require complete continuity of radial and tangential stresses and vertical and radial displacements along the cylindrical boundary between materials. The test conditions require that the entire system be under a uniform displacement. The formulation of the strain problem is shown in Figure 19.

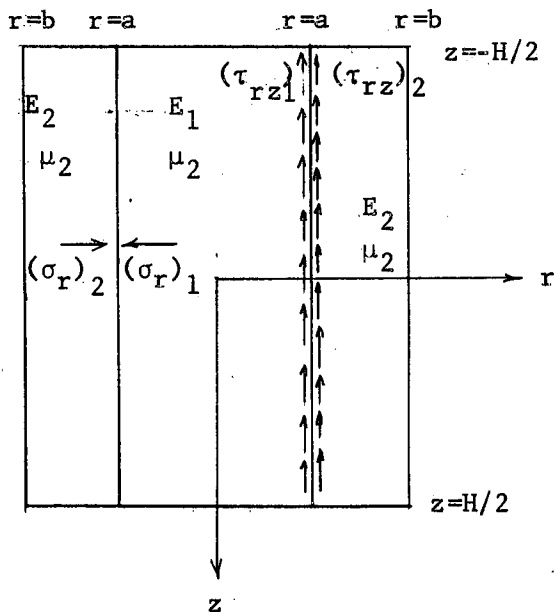


FIGURE 19

TWO-PHASE MATERIAL PROBLEM

In terms of a one dimensional simplification, the stress picture results from a condition of equality of strain across the entire cross-section. Since the outer material is hard, the resulting stresses will be larger than in the soft inner core. At a given level of strain the outer ring will fail and the softer inner material will be in a pre-failure state. The outer failure will be in the usual manner. Once the outer skin has ruptured, the tendency will be for the bond between the two phases to be broken and for the inner portion to remain in compression. However, since the outer skin does not completely remove itself, the net effect is to impose lateral strain restraints on the soil, increasing its strength and altering the character of the failure surface. A photograph of the failure of a partially dried specimen is shown in Figure 20.

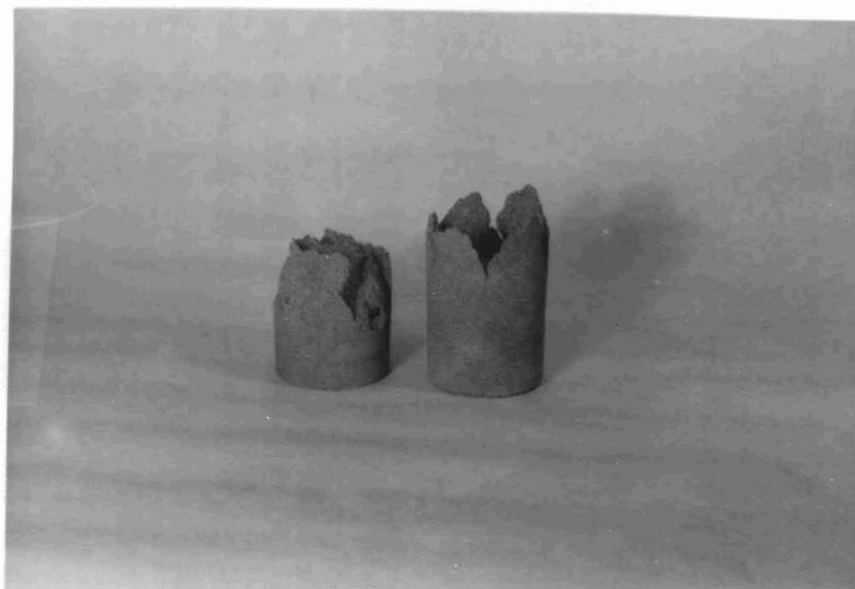


FIGURE 20

TWO-PHASE FAILURE

The relationship between time of curing and strength increase of the soil as shown in Figure 13, indicates that at an early age the strength actually drops off. This is an apparent phenomena and is not truly indicative of the inherent soil strength. The stresses computed are based on the overall sample diameter. At an early age the outer skin is so thin that it is of little consequence in restraining the sample laterally. In actuality, the true area is less than the nominal area for these early stages of drying, and thus the indicated early result is lower than the initial strength.

The strength-time relation in Figure 13, for ages after 15 days is in question, due to the lack of control tests run on these samples. For this reason the drying experiment is being repeated. The results of this repeat experiment will be reported at a later date in a supplementary report.

3. EFFECT OF WET-DRY CYCLES

The results of the wet-dry experiment as shown in Figure 14, indicate the trend of strength reduction due to alternate wetting and drying. The results in Figure 14 are based on an average initial strength in which the effects of relative density are averaged out. If the soil is dried for a short period and then rewet, there is a finite reduction in strength and this reduction increases with the increase in length of drying and is further influenced by cycling until a stable reduction is achieved. The maximum reduction in strength is approximately one-half of the freshly stabilized strength.

There are two basic influences to consider. The first of these is the influence of the drying period on the strength after wetting, while the second is the influence of wet-dry cycles on the strength for short drying periods. When the sample dries, the chemical bonds are stretched and broken. Considering only the effects of chemical bonding (eliminating capillary stresses), the longer the drying process, the potentially weaker the sample will become. Thus, on rewetting when the capillary stresses are relieved the polymer will have lost much of its strength. It is therefore logical that the rewet strength should be inversely proportional to the length of drying up to a point where complete equilibrium is achieved in the extensional rupture of chemical bonds. When this point of equilibrium is achieved, all the bonds that are likely to rupture will have ruptured, and there will be no effect of cycles of wetting and drying. Prior to this point, however, the greater the number the cycles, the lower the strength again up to a equilibrium number of cycles. The influence of cycling is similar to a fatigue process, in which bonds are strained and then relieved alternately until they reach a condition of failure. The equilibrium position is one

in which the weaker bonds are broken and the cycling will not influence the stronger bonds, as in a fatigue limit. The relative effects of the drying period is of greater importance than the effect of cycles of wet and dry, simply due to the magnitudes of the effects involved.

A minor study was made of the effects of relative density on wet-dry cycles strength. In the laboratory work it was impossible to attain the same relative density for each sample. It was possible, based on this work and previous work⁽¹⁾, to develop a statistically mean relation between the strength decrease and the relative density over the limited range of relative densities used. It was qualitatively found that the strength ratio decreased with an increase in relative density. After the strength equilibrium point has been achieved, the rewet strength appeared to be independent of the relative density. Thus the strength of the denser samples would show a greater decrease than the less dense samples. An increase in density of a soil will portend a decrease in the capillary radius during a given drying stage. This means that the capillary forces will be higher and the stresses in the gel will be greater than before. Thus more bonds will fail and the strength of the gel will tend to reduce. This reduction is balanced by the increase in strength due to an increase in relative density which will result in a net rewet strength that is independent of relative density.

The typical density change curves of Figure 10a thru 10f, indicate a single general trend, namely that at the density at the end of drying is reduced as the number of previous cycles increases. This reduction is greater than the reduced increase due to wetting which indicates that after the initial drying the range of fluctuation of density is increasing. This fact is nothing more than the measure of the free

water held in the gel and the soil voids after the initial drying. After the first drying period the wetting fills the voids formed and a relatively small amount of water is absorbed by the gel. The subsequent drying drives out the void water and along with it more of the gel water than had been previously evaporated. With rewetting again the voids were filled and the weight gain increased.

In terms of pilot experimentation, with a single soil, it would seem that an extension of this work would be most valuable in terms of relative density. The experiment that was undertaken used a two parameter model, the attributes being, length of cycle and number of cycles. The useful extension would be to add an attribute of relative density, in which this variable was tested on two levels, namely the loose and the dense state.

E. CONCLUSIONS

The conclusions that can be drawn concerning this study of the mechanical behavior of a granular soil when treated with AM-955, and subsequently aged and rewet are:

(1) The age and storage conditions of the raw catalyst system will influence the stability of the polymerized gel for percentages of AM-955 less than ten.

(2) The presence of trace quantities of common soil minerals other than silica will profoundly influence the time of gellation.

(3) There will be no noticeable volume change in fully saturated soils subject to axially symmetrical evaporation surfaces.

(4) Drying of soil stabilized with AM-955 will produce an increase in strength which will be 9 or more times the freshly gelled strength.

(5) The increase in strength due to drying will be due to the imposition of capillary forces.

(6) Drying of stabilized soil will proceed in layers with the rate of drying varying between outer and inner layers, the outer layers drying at the fastest rate.

(7) The rate of drying will be dependent on the surface area available to evaporation at a given instant and the vapor pressure differential across a free boundary. The rate of drying is inversely proportional to the surface area available.

(8) Drying follows an exponential decay law in which the volume and surface area influence the frequency of the exponential.

(9) Failure of partially dried stabilized soil by straining will initiate in the dry portion at small strains and propagate to the wet area of the soil as modified by the constraining effects of the dry area.

(10) Extended time of drying and cycles of wetting and drying will reduce the wet strength as much as 1/2 of the strength of the freshly stabilized soil for soils in the dense state.

(11) To a point of strength equilibrium, the rewet strength is inversely proportional to the length of drying.

(12) For short drying periods the rewet strength is inversely proportional to the number of cycles of wetting and drying.

(13) For long drying periods the rewet strength is independent of the number of wetting and drying cycles.

(14) Successive equal wetting and drying cycles will result in greater density reductions at the end of the drying period as the number of cycles increases.

(15) The effect of length of drying on strength reduction is relatively greater than the effect of number of wet-dry cycles.

(16) In the dense state the equilibrium strength of the rewet soil is independent of the relative density.

(17) In the dense state the ratio of the rewet strength to the initial strength is reduced with increases in relative density.

F. ACKNOWLEDGMENTS

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