

STABILIZATION OF CLAY WITH
4-TERT-BUTYLPYROCATECHOL

By

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1962

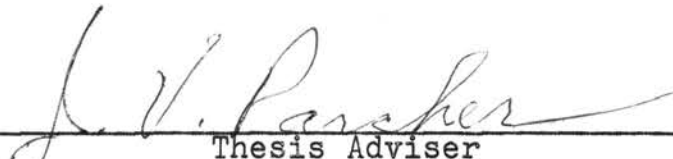
Submitted to the Faculty of the Graduate School
of the Oklahoma State University
in partial fulfillment of
the requirements for
the degree of
MASTER OF SCIENCE
August, 1964

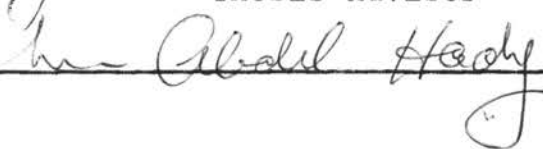
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569568

ACKNOWLEDGEMENTS

The writer wishes to acknowledge his indebtedness to some of the people who have made his graduate education possible.

To Professor James V. Parcher, for his guidance and encouragement throughout my graduate education and for his assistance in the preparation of this thesis.

To Professor Jan J. Tuma, Head of the School of Civil Engineering, and all others responsible for my being awarded a Graduate Assistantship.

To my parents, Mr. & Mrs. Joseph T. Greco, for their encouragement and support throughout my education.

To my wife, Davene, for her understanding and encouragement during my graduate education, and for her typing of this thesis.

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CHAPTER I

INTRODUCTION

General

A question of importance throughout the years has been how to construct long-lasting, durable highways at a minimum of cost. One aspect of this problem is the selection of the materials to be used in the construction of the structural components of the pavement. In some areas of the United States, this problem is economically solved by the use of selected aggregates from nearby borrow areas. In other sections of the United States, borrow areas may be non-existent or else have become depleted due to prior construction. If such is the case, the highway engineer must resort to other techniques or procedures to adequately complete the task at hand.

One solution to the problem is soil stabilization. Originally, soil stabilization, as the name implies, meant primarily the increase in the strength of soil. Today, soil stabilization encompasses all techniques used in the alteration of any engineering property of the soil.

Origin

Research to develop new techniques of soil stabilization is continuing today with additional emphasis on the use of chemical additives. One such chemical additive, 4-Tert-Butylpyrocatechol, was tested for use in the stabilization of the Permian Red Clays of Oklahoma. The chemical 4-Tert-Butylpyrocatechol is commercially produced by the Dow Chemical Company under the trade name of Terbec, and will be referred to as such throughout this thesis.

Extent of Investigation

Under the direction of Professor James V. Parcher of the School of Civil Engineering at Oklahoma State University research was initiated to determine what effect Terbec had on the Oklahoma clays. The effect of Terbec on typical soil types has been studied elsewhere (1). The results of these initial studies show that optimum curing conditions for the Terbec-soil mix is seven days air drying at a relative humidity of approximately 50 percent. Due to this prior research with Terbec, a considerable savings in time and energy was realized, thereby, resulting in a more specific and extensive testing on treatment rates.

Research in the present investigation was limited to one particular soil type which is assumed to be representative of the so-called problem soils of Oklahoma. These problem soils exhibit loss of strength and volumetric instability in the

presence of a plentiful supply of water. Terbec alters the adverse characteristics of these clays and renders them construction materials.

The treatment rate for a given soil is dependent upon the mineralogical composition of that soil. Therefore, for soils which are not typical, a limited amount of testing is essential in order to optimize the treatment rate. For the clay type tested, a treatment rate of 0.20 percent or more is necessary for effective subgrade stabilization.

CHAPTER II

CLAY MINERALOGY

Introduction

Clay is a term used to describe a natural, earthy, fine-grained material which develops plasticity when combined with a limited amount of water (Grim 2). By plasticity is meant the ability of a soil to undergo change in shape without rupture, volume change, or elastic rebound.

For engineering purposes, soils are sometime classified on the basis of the grain size of the constituent particles. A number of such classification systems exist, and each individual system has its own arbitrary method of classification. Clay is defined by many of these systems as those soil particles which have a grain size less than 0.002 mm. The other classification systems use other arbitrary limits to differentiate between the clay and not-clay. These systems are all deficient in one respect; some soils which fall within the range of clay sizes may not be composed of clay minerals and subsequently should not be called clay.

According to Grim (3), clay minerals are extremely small particles having a crystalline structure and which are the hydrated silicates of aluminum, magnesium, and iron. There

are two basic units or building blocks of which the clay minerals are composed: the silica tetrahedron and the octahedral aluminum hydroxide. The clay minerals are divided into three general groups according to the arrangement of these two basic units: the kaolin group, the montmorillonite group, and the illite group.

Atomic Structure

An atom is analagous in some respects to our own planetary system. Electrically charged particles called electrons revolve about a nucleus in concentric paths or orbits. Each element has a discrete number of electrons which distinguishes it from other elements. The orbits surrounding the nucleus are such that the first orbit may contain no more than two electrons, whereas, the other orbits may contain as many as eight. An orbit will not contain an electron unless all the inner orbits are filled to capacity. When the number of electrons in the outermost orbit is less than eight, the atom is chemically active and may combine with other atoms to form molecules and solids. These electrons in the outermost orbit are called valence electrons.

When an atom loses or gains electrons, the atom as a whole is left in an unstable state. This ability of an atom to lose or gain electrons enables it to combine with similar or different atoms.

Interatomic Bonds

The bonding of atoms to one another is accomplished by one of three distinct types of bonds: covalent, ionic, or metallic.

A covalent bond results from the sharing of pairs of valence electrons by two or more atoms. An example of a covalent bond is that between two oxygen atoms in an oxygen molecule.

An ionic bond is formed when atoms of different elements transfer electrons one to the other so that both have stable outer orbits. An example of such a bond is that between sodium and chlorine. Here the sodium atom gives up its one valence electron to the chlorine atom thereby completing its outermost orbit. The union between these two atoms results in the molecule sodium chloride.

In the metallic bond atoms of the same or different elements give up their valence electrons to form an electron cloud throughout the space occupied by the atoms (Richards 4). When the atoms give up their valence electrons they become positive ions, which are attracted electrostatically to the negatively charged electron cloud.

Intermolecular Bonds

A molecule is the smallest entity that retains the chemical properties of the original material. The forces that hold molecules together are considerably weaker than

atomic bonds and therefore are referred to as secondary bonds. There are three known types of intermolecular bonds: dispersion, dipole, and hydrogen.

Dispersion bonds are generally formed between molecules consisting of one atom. The electrons of these atoms revolve in such a way that they become oscillating dipoles and subsequently result in a net attractive force between atoms.

Dipole bonds are formed between molecules which are permanent dipoles. Hydrogen chloride, for example, is a covalent-bonded molecule in which the hydrogen atom has a small positive charge and the chlorine atom has a corresponding negative charge. Hydrogen chloride molecules are therefore bonded between oppositely charged ends.

The hydrogen bond is essentially a special type of dipole bond, but one that is much stronger. It occurs when the loosely held valence electron of hydrogen is given up to the adjacent atom in the molecule. There results a permanent dipole which is attracted to similar dipoles. A typical example of the hydrogen bond is water or ice.

Basic Components

There are two basic components of which all clay minerals are composed. The first unit or building block is the silica tetrahedron. A silica tetrahedron consists of a central silicon atom surrounded by four oxygen atoms that are equidistant from the silicon atom (Grim 5). A number of such tetrahedra joined by ionic and covalent bonds forms a

sheet structure in which the base plane of oxygen atoms is shared by adjacent tetrahedra (Means and Parcher 6).

The second building block consists of two sheets of oxygen or hydroxyls in which aluminum, iron, or magnesium atoms are embedded in an octahedral arrangement, so that they are equidistant from six oxygens or hydroxyls. When aluminum is present, the structure is called gibbsite. When these octahedral units are joined in a sheet structure, they will combine in different arrangements with the sheet silica tetrahedra to form the clay minerals.

Primary Clay Minerals

There are three primary clay mineral groups: kaolinite, montmorillonite, illite. According to Means and Parcher (7), kaolinite ($\text{Al}_2 \text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$) is derived from the chemical weathering of feldspar and other aluminum bearing rocks. The form element of kaolin is composed of one silica sheet and one gibbsite sheet bound together at the apex of the tetrahedra by ionic and covalent bonds. Elementary kaolin particles are composed of form elements of kaolin stacked one on top of the other and joined by the hydrogen bond.

Montmorillonite ($\text{Al}_2 (\text{O}_{3.5}\text{SiO}_2 \cdot 0.5-7\text{H}_2\text{O})$) is composed of units made up of two silica tetrahedral sheets with a central alumina octahedral sheet. When montmorillonite particles are formed, the form elements are joined such that the silica tetrahedra are in contact, with the result that a weak bond is formed (Grim 8). Consequently, water molecules can enter

between montmorillonite sheets in varying amounts giving montmorillonite an expanding lattice as opposed to the fixed lattice of kaolinite and illite.

Illite is similar in structure to montmorillonite except that potassium ions occupy positions between the adjacent silica layers.

Ion Exchange

Grim (9) states that clay minerals have the property of adsorbing certain cations and anions which can be exchanged by other cations and anions in an aqueous solution. These exchangeable ions are held around the clay mineral but they do not affect the structure of the mineral itself. The ability of a clay to adsorb ions on its surfaces or edges is a function of the mineral structure of the clay and the size of the particles.

Cation or base exchange capacity of a soil describes the ability of a soil to adsorb exchangeable cations. Base exchange capacity is measured in milliequivalents per 100 gm of dry soil. One milliequivalent is one milligram of hydrogen or the portion of any ion which will combine with or displace one milligram of hydrogen. The cation-exchange capacity in milliequivalent per 100 grams of three common clay minerals is: montmorillonite----80-150, illite----10-40, kaolinite---3-15.

Anion exchange phenomena are not well understood due to the decomposition of the mineral structure during the reacting

process.

Clay-Water Relations

Clay minerals as a whole are electrically neutral, but due to the arrangement of the atoms, the surfaces of these particles are charged negatively. Water molecules, being permanent dipoles, attach themselves to the clay particles. More than one layer of water molecules can be adsorbed in this manner, but as the distance from the surface increases the attractive force holding the water molecule decreases.

This adsorbed water near the surface is referred to by some investigators as not-water. The term not-water is used because the adsorbed water does not have the properties of normal water and because a name for it has not yet been agreed upon. Whatever the name given to it, this not-water significantly affects the behavior of clay particles when they are subject to an applied stress, since the adsorbed water layers separate the clay particle surfaces.

CHAPTER III

CHEMICAL SOIL STABILIZATION

Definitions

Soil stabilization, as defined by Winterkorn (10), includes every physical, physico-chemical, and chemical method employed to alter any property of soil to improve its engineering performance. According to Murray (11), soil stabilization processes can be grouped into two separate categories, physical and chemical. For the sake of simplicity, chemical stabilization can be defined as all techniques in which the final stabilized product is the result of the combination of the soil with a chemical. In addition, the chemical additive must produce a reaction which changes the inherent properties of the soil. All other procedures, such as compaction, bituminous stabilization, and portland cement stabilization, will be referred to as physical stabilization.

The number of chemicals available to the highway engineer is seemingly endless. Which chemical is the best for a given situation is a question that cannot be answered simply. Therefore, the highway engineer must resolve this problem with the aid of the experiences of others, but

tempered by his own experience and judgement.

Chemical Reactions

When a chemical is incorporated into a soil for the purpose of stabilization, a chemical reaction must take place. There are numerous chemical reactions, but as yet, only four have proved to be applicable in the soil stabilization field (Murray 12).

- (1) Ion Exchange - consist of the replacement of the ion attached to the soil particle by a different ion. This is particularly important in the stabilization of clays, since many of the properties of clays are related to the exchangeable ion.
- (2) Precipitation - is accomplished by the combination of two chemicals which will react to form a gel. A common example is the combination of sodium silicate and calcium chloride to form the insoluble compound calcium silicate.
- (3) Polymerization - is induced in the soil mass by the addition of two simple compounds which will react with each other to form very large molecules. These large molecules are synthetic resins which act as a filler material in the soil and the final stabilized soil is a kind of plastic. Common examples are anilin-

furfural and calcium acrylate.

- (4) Oxidation - is said to account for some stabilization reactions, but to date no research to substantiate this fact has been presented.

A fifth reaction has been postulated by Hemwall (13) to account for the stabilizing effect of Terbec. Hemwall states that the adsorption of Terbec is via the formation of a surface aluminum-catechol complex, and that the rate of this reaction is determined by the rate of clay lattice decomposition to yield aluminum.

Chemical soil stabilizers can also be classified (Sherwood 14) as to how the strength of the stabilized soil is maintained or produced. These three categories are bonding agents, water proofing agents, and combination bonding or waterproofing agents. Bonding agents stabilize soils by creating physical and/or chemical bonds between the soil particles which enhance the strength of the soil under both wet and dry conditions. Waterproofing agents do not affect dry strength significantly but allow the dry strength of the soil to be retained in the presence of water by reducing water absorption.

Lime

Lime is used for the stabilization of bases, sub-bases and subgrades in climates where freezing and thawing is not a problem. In Oklahoma, lime is being used for the stabilization of subgrades which are composed of the problem soil

types. Also, some subgrades, which were not previously stabilized with lime, have received this treatment by injection through the pavement slab to counteract the instability of these subgrades. Recent reports indicate that these subgrades are now functioning properly.

When lime is mixed with moist soil, three types of reactions occur. The first reaction occurs when calcium ions, in the lime, are exchanged for ions on the clay particle surface resulting in a decreased electric charge between particles. This decreased charge results in particle coagulation which causes a reduction in the plasticity of cohesive soils. The second reaction which occurs is the carbonation of the lime by the carbon dioxide in the air. This reaction should be prevented because the lime will not be as effective as a soil stabilizer. The final reaction, called pozzolanic reaction, results in a cementation of soil particles. This reaction occurs between the lime and the alumina and silica which are components of the soil. Fly-ash, having a high silica content, is sometimes added to the soil to further induce pozzolanic reaction. Also, the addition of some sodium compounds will improve the strength of the soil-lime mix (Lambe 15). In general, lime increases the strength of almost all types of soils. For granular soils lime treatment levels from two to five percent are usually required, and for plastic soils treatment levels from five to ten percent are normally required.

Gypsum

Research, at Oklahoma State University (16, 17, 18, 19, 20) has indicated that the addition of gypsum alone will not economically stabilize the clay soil of Oklahoma. However, gypsum does lower the plasticity of the clay and its swelling potential. This has been explained as a physical rather than chemical phenomenon.

If a trace chemical, such as sodium hydroxide, is added to the clay-gypsum mix at a treatment rate of two percent by weight, the final product can be used for the stabilization of highway subgrades. Additional field testing is necessitated before final judgment can be levied as to whether gypsum and sodium hydroxide should be used for stabilization work.

Chlorides

Sodium chloride and calcium chloride have been used as dust palliatives for more than thirty years. These salts are generally used for the construction of granular surfaces and base courses. The effect these salts have on soils is two-fold; they alter the characteristics of the soil water and they cause colloidal reactions to occur.

The primary attribute of the salt is in the changes it makes in the properties of the water in the pores. These changes reduce the loss of moisture from the soil and are explained by the fact that the salts are deliquescent and

hygroscopic and lower the vapor pressure of the water. Salt also reduces or prevents frost heave in soil by lowering the freezing point of water.

A problem in the use of these chlorides is how long they will last. The performance of salts is dependent upon whether they will wash out from the soil pores. The use of an impervious surfacing will prevent infiltration and therefore reduce leaching.

Lignin

Lignin is a waste product produced by the papermaking industry. Lignin is extracted from wood by treatment with sulfite chemicals resulting in calcium lignosulfonic acid. This acid is then treated with lime to give calcium lignosulfonate, sometime called ligninsulfonate or even lignin. About 3 million gallons of calcium lignosulfonate are produced in the United States and Canada every day, and therefore constitutes a major waste disposal problem. A means of utilizing this waste was found to be in the stabilization of soils.

Lignin acts primarily as a binder, cementing soil particles together and greatly increasing the dry strength of the soil. Lignin also acts as a dispersion agent, resulting in a lower permeability of the soil. This lower permeability accounts for the reduction in the frost heaving of the soils treated with lignin.

Since lignin is water soluble, its stabilizing effects

are not permanent. To rectify this problem, sodium bichromate or potassium bichromate were mixed with lignin and the resultant product was an insoluble gel. This process is called the chrome-lignin process.

4-Tert-Butylpyrocatechol (Terbec)

It is a known fact that fine-grained soils possess considerable strength when they are dry. However, upon access to water, they will lose some or all of their strength. For example, a dry sample of Oklahoma Permian clay has an unconfined compressive strength of approximately 35 kg/cm^2 , but will completely disintegrate upon immersion in water. In an attempt to reduce the adverse effect of water on these soils, trace chemical waterproofers were developed which retain the dry strength of soils. These chemicals do not bond the soil particles, but just change the soil from hydrophilic to a hydrophobic state.

Hemwall and co-workers (1) have studied the effect of Terbec on various soil types. From their laboratory studies they have found that Terbec is an effective stabilizing agent for some soil types. Terbec is applied to soil as an emulsion in the molding water. The molding water is that amount of water necessary to bring the soil up to its compaction moisture content. The soil is then compacted by conventional construction procedures. The quantity of Terbec is expressed as a percentage of the maximum dry density. The soils that react with Terbec most favorably are silty in

nature and contain between 10 percent and 40 percent clay. Treatment rates between 0.05 percent and 0.10 percent are necessary to stabilize these soils. Optimum curing conditions are found to be air drying for seven days at a relative humidity between 30 and 50 percent. The testing procedure used in Hemwall's (1) work was, after curing the samples for seven days, to immerse the samples for 24 hours in distilled water and then measure the unconfined compressive strength.

Other Chemicals

Many other chemicals have proved to be acceptable as soil stabilizers and a few of the more important ones will be mentioned for the sake of completeness. The chemicals to be mentioned can be grouped into two categories, synthetic resins and inorganic compounds.

Synthetic resins are organic solid or semisolid materials produced by the union of molecules into large polymers. The synthetic resins encase the soil particles and bond them together. The polymerization of aniline and furfural in a soil results in a plastic soil mass that is very well stabilized. The cost of these chemicals is not competitive with other stabilizers and they are not used except in cases where stabilization is needed in a few hours.

Calcium acrylate polymerizes with certain catalysts to form a hard rubbery stabilized soil mass. This polymerization is only temporary and upon the addition of water the

stabilized soil becomes very soft. The use of calcium acrylate is restricted only to unusual situations.

Phosphoric acid is an inorganic compound which reacts with calcium or other cations in a clayey soil to produce insoluble phosphates which behave as a cement. Two percent additive is considered to be economical. This chemical is now in the field testing stage.

CHAPTER IV

MATERIALS AND TESTING PROCEDURES

Permian Red Clay

The clays of the Permian period are marine clays that were deposited on the bed of an inland sea that covered large portions of Oklahoma. The Permian clay was selected for study because of its widespread occurrence and troublesome nature. The physical characteristics of these clays, as with many natural occurring soil deposits, are of a highly variable nature; therefore, all statements made concerning these clays are general in nature.

Many of the Permian clays contain sand and/or silt, while others contain no appreciable amounts of either. These clays contain approximately 40 percent montmorillonite, 30 to 35 percent illite, and 25 to 30 percent unidentified material. The unidentified material is believed to be quartz, kaolinite and iron oxide. Because of the expanding lattice of montmorillonite, even small percentages of this mineral greatly influence the physical properties of the Permian clays. For clays containing a large percentage of montmorillonite, the liquid limit is generally never less than 100 percent. Clays which contain large percentages of sand and silt

have a liquid limit of approximately 35 percent. For the Permian clays in general, the liquid limit is, on the average, about 50 percent and the plastic limit is about 15 percent.

The Permian clay used in this investigation was obtained from the basement of the Life Sciences Building on the Oklahoma State University campus at Stillwater, Oklahoma. The clay was pulverized and passed through a number ten sieve. The initial water content was determined to be 3.8 percent. By standard procedures the liquid limit and plastic limit were found to be 31.6 percent and 11.0 percent, respectively. A Harvard Miniature Compaction Test yielded a maximum dry density of 112.5 pcf at an optimum moisture content of 17 percent. A grain size distribution curve of this clay is shown in Figure I.

4-Tert-Butylpyrocatechol (Terbec)

The chemical 4-Tert-Butylpyrocatechol is commercially produced by the Dow Chemical Company under the trade name of Terbec. A sample of Terbec was sent to Professor James V. Parcher by R. A. Helmer of the Oklahoma Highway Department. Mr. Helmer had received this sample from Harvey H. Shafer of the Dow Chemical Company. The results of the tests with Terbec are presented in the following chapter.

Chemically speaking, a more descriptive name for Terbec is 1,2 - dihydroxyl - 4 (2-methyl) - propylbenzene. The arrangement of the atoms in the structure of a molecule of Terbec is shown in Figure 2.

Due to the toxicity of Terbec, precautions should be taken to avoid any contact upon the skin of concentrated Terbec. In case of accidental contact with concentrated material, Terbec should be quickly and thoroughly removed from the skin; washing the skin with soap and water is a recommended procedure.

Preparation of Samples

The air-dried clay that had passed the number ten sieve was combined with a mixture of Terbec and molding water; Terbec was an emulsion in the molding water. The molding water is that amount of water necessary to bring the soil up to its compaction moisture content. The soil-water-Terbec combination was mixed by hand until a uniform consistency was obtained.

The amount of Terbec added to the molding water was based on a percentage of the maximum dry density of the soil. For example, at a treatment rate of 0.2 percent for 750 gm soil sample air-dried to an initial water content of 3.8 percent, the amount of Terbec was calculated to be 1.44 ml.

The soil specimens were compacted using the Harvard Miniature compaction equipment designed by S. D. Wilson in 1949. This equipment consists of a mold 1.312 inches in diameter and 2.816 inches in length. The mold and its extension collar are held to a base and compaction is accomplished by means of a tamping force utilizing a spring loaded plunger. The compacted samples are extruded from the

mold by means of a special device. In this investigation the soil was compacted in three layers, each layer being subjected to 25 repetitions of a 20 pound force from the tamper.

The advantages to using the miniature compaction test are:

- (1) Only a small quantity of soil is needed to provide the required results.
- (2) The kneading action of the miniature compaction test more closely resembles the action of sheepsfoot roller than does the dynamic action of most standard compaction tests.

The disadvantages to using the miniature compaction tests are:

- (1) The results of these compaction tests have not been correlated completely with the standard compaction tests.
- (2) Its use is restricted only to soils passing the number ten sieve.

After extrusion of the soil samples from the mold, they were allowed to air dry at a relative humidity of approximately 50 percent for 7 days. Subsequently, they were immersed in distilled water for 24 hours and then were subjected to the unconfined compression test.

Unconfined Compression Test

The unconfined compressive strength test is one of the

simplest and most widely used laboratory strength tests of cohesive soils. The results of these tests have been correlated extensively with the actual strength of the soil in place. Because of the confinement of the soil in the ground, the failure condition in the field is considerably different from the failure of the soil in the laboratory. However, the strength of the laboratory specimens can be used as a comparison of the in place strength of the soil.

Two types of unconfined compression test equipment were used in this investigation. The first type used was such that the loading rate was controlled and the strain for a particular loading was measured; the loading rate used was 10 grams per minute. In the device used, the axial load was applied by means of a horizontal bar that is drawn downward by means of a jack; a platform scale measures the load applied to the specimen. An Ames dial indicator was used to measure the deformation of the sample.

The second type of equipment used was a Karol-Warner Model 550 Unconfined Compression machine equipped with a Model RW-38 chart recorder. The machine is driven by a 1/6 th hp. electric motor whose speed is controlled by a Model KWDV-2 Variac which allows the strain rate to be controlled; the strain rate used was 0.25 in./min. This apparatus was mainly used as a check of the accuracy of the first type of equipment.

CHAPTER V

TEST RESULTS

Initial Studies

Five samples of the Permian clay, without any chemical additive, were compacted at their optimum moisture content. These five specimens were air-dried for seven days at a relative humidity of approximately 50 percent. Four of these specimens were brought to failure at an average unconfined compressive strength of 35 kg/cm². The fifth sample was immersed in distilled water. Within 15 minutes after immersion, this Permian clay sample completely disintegrated.

When Terbec was added to the soil as an emulsion in the molding water, the deleterious effects of water on the clay samples was reduced at all treatment levels. For all treated soil samples tested in this study the specimens were compacted at their optimum moisture content and then cured for seven days at a relative humidity of approximately 50 percent. They were then immersed in distilled water for twenty-four hours. Within ten minutes after being removed from the distilled water they were brought to failure in an unconfined compression test.

Low Treatment Rates

At treatment rates of 0.15 percent and lower, the strength of the treated soil mass was not measurable. Terbec reduced, to a considerable degree, the susceptibility of the specimens to slaking, but as the samples were being removed from the water they literally fell apart.

At the initial stage of this study, eight samples, four at a treatment rate of 0.10 percent and four at treatment rate 0.15 percent were prepared. These eight specimens had zero unconfined compressive strength. Two months later, eight more samples at treatment rates of 0.10 percent and 0.15 percent were prepared and these samples exhibited the same characteristics as the eight compacted previously.

High Treatment Rates

As the treatment level was increased above 0.15 percent, the condition and strength of the soil specimens improved. For this phase of the study, the treatment rates used were 0.20 percent, 0.25 percent, and 0.50 percent. As was noted before, all soil samples were compacted, cured, and tested in the same manner.

Initially, fifteen samples were prepared, five specimens each at rates of 0.20 percent, 0.25 percent, and 0.50 percent. The first fifteen samples and fifteen of the twenty-seven subsequent samples were brought to failure via the controlled loading rate unconfined compression test.

The other twelve of the twenty-seven were tested with the controlled strain rate unconfined compression machine. This procedure was adopted so that the results from the first fifteen samples could be checked with results of fifteen other similar samples prepared at a later date. The other twelve samples enable a correlation to be made between the controlled loading rate and the controlled strain rate at the three high treatment levels.

At a treatment level of 0.20 percent, the average unconfined compressive strength of both groups of five samples tested at a controlled loading rate was 0.80 kg/cm^2 at a strain of 7.0 percent. Figures 3 and 4 show the stress-strain curves for a representative sample from the first and second groups of five samples, respectively. The average unconfined compressive strength of the four samples tested at a controlled strain rate was 0.90 kg/cm^2 .

At a treatment rate of 0.25 percent, the average unconfined compressive strength of both groups of five samples tested at a controlled loading rate was 0.95 kg/cm^2 at a strain of 6.8 percent. Figures 5 and 6 show the stress-strain curves for a representative sample from the first and second groups of five samples, respectively. The average unconfined compressive strength of the four samples tested at a controlled strain rate was 1.15 kg/cm^2 .

At a treatment rate of 0.50 percent, the average unconfined compressive strength of both groups of five samples tested with controlled loading was 1.20 kg/cm^2 at a strain

of 5.7 percent. Figures 7 and 8 show the stress-strain curves for a representative sample from the first and second groups of five samples, respectively. The average unconfined compressive strength of the four samples tested at a controlled strain rate was 1.90 kg/cm^2 .

These results indicate that the unconfined compressive strength from the controlled loading rate test is lower than the strength obtained from the controlled strain rate test. A possible reason for this is that the controlled loading rate test took longer to run than did the controlled strain rate test and therefore would give a lower value for the strength.

The test results also show that a treatment rate greater than 0.50 percent would increase the strength of treated soil. This means that the optimum treatment rate for the Permian clay may be economical rather than physical; higher treatment rates would give higher strengths.

CHAPTER VI

CONCLUSIONS

General

This study was initiated for the purpose of evaluating the effectiveness of Terbec as a stabilizing agent for the Permian Red clays. The unconfined compression test, using both a controlled loading rate and controlled strain rate, was used as a measure of the strength. All samples were prepared and tested as follows:

- (1) Compaction with Harvard Miniature Compaction apparatus at an optimum moisture content of 17 percent.
- (2) Air drying of the samples for seven days at a relative humidity of approximately 50 percent.
- (3) Immersion in distilled water for twenty-four hours.
- (4) Subjection to unconfined compression test.

From this investigation the following general conclusions can be formulated:

- (1) Terbec will reduce the deleterious effects of water on the Permian clays at all treat-

ment levels; the higher the treatment rate the better the condition of the sample.

- (2) Treatment rates of Terbec equal to or greater than 0.20 percent are necessary in order that effective subgrade stabilization can be realized.
- (3) The optimum treatment rate for the Permian clays is based on the economics of the problem not on the physical combination between Terbec and the Permian clays; higher treatment rates of Terbec will give higher strengths for the Permian clays.

Specific

It was not the purpose of this study to conclude that a specific treatment rate is suitable for all the Permian clays. Such a conclusion would not be feasible due to the highly variable nature of the Permian clays. Instead it is recommended that a limited amount of testing for the soil in question, be carried out so that an economical treatment rate can be found.

Further research with Terbec for the Oklahoma clays could be initiated for the purpose of obtaining a relationship between treatment rates of Terbec and physical characteristics of the clays. If this were done, it would be possible to tell immediately what treatment rate of Terbec would be needed to stabilize a particular soil and also whether this rate would compete economically with other stabilizing agents.

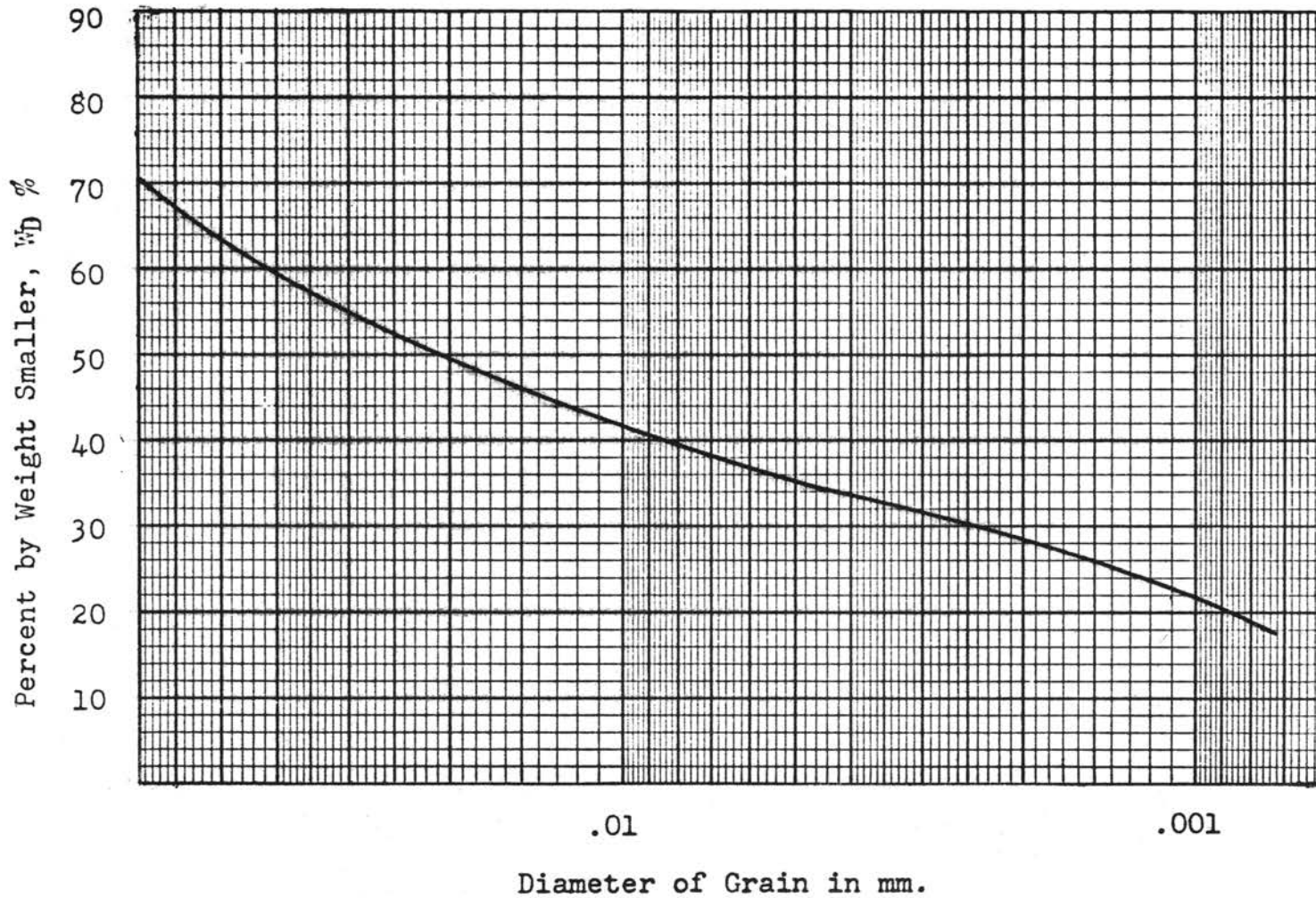


Figure 1
 Grain Size Distribution Curve
 For Permian Red Clay

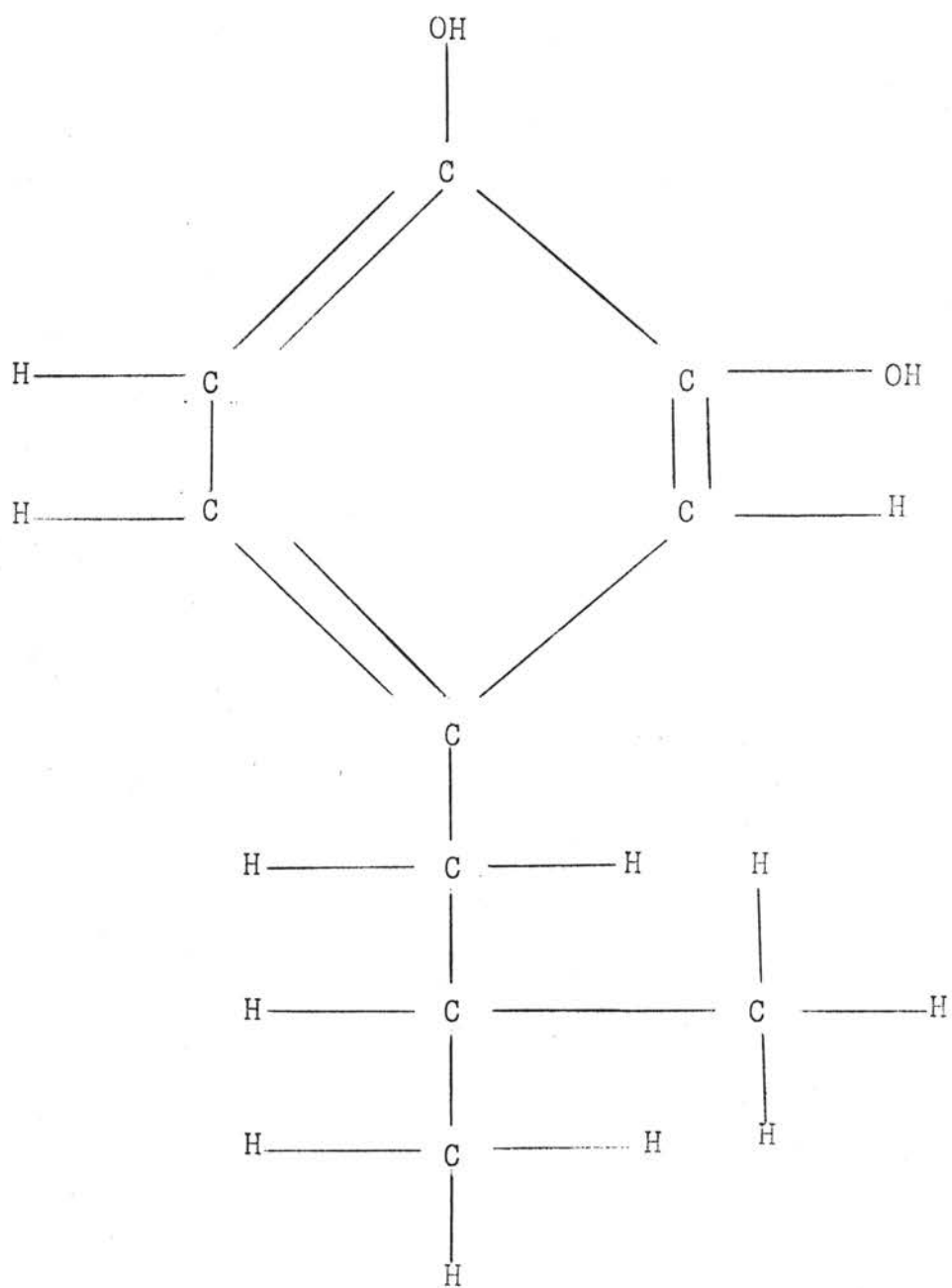


Figure 2
Arrangement of Atoms in
Molecule of Terbec

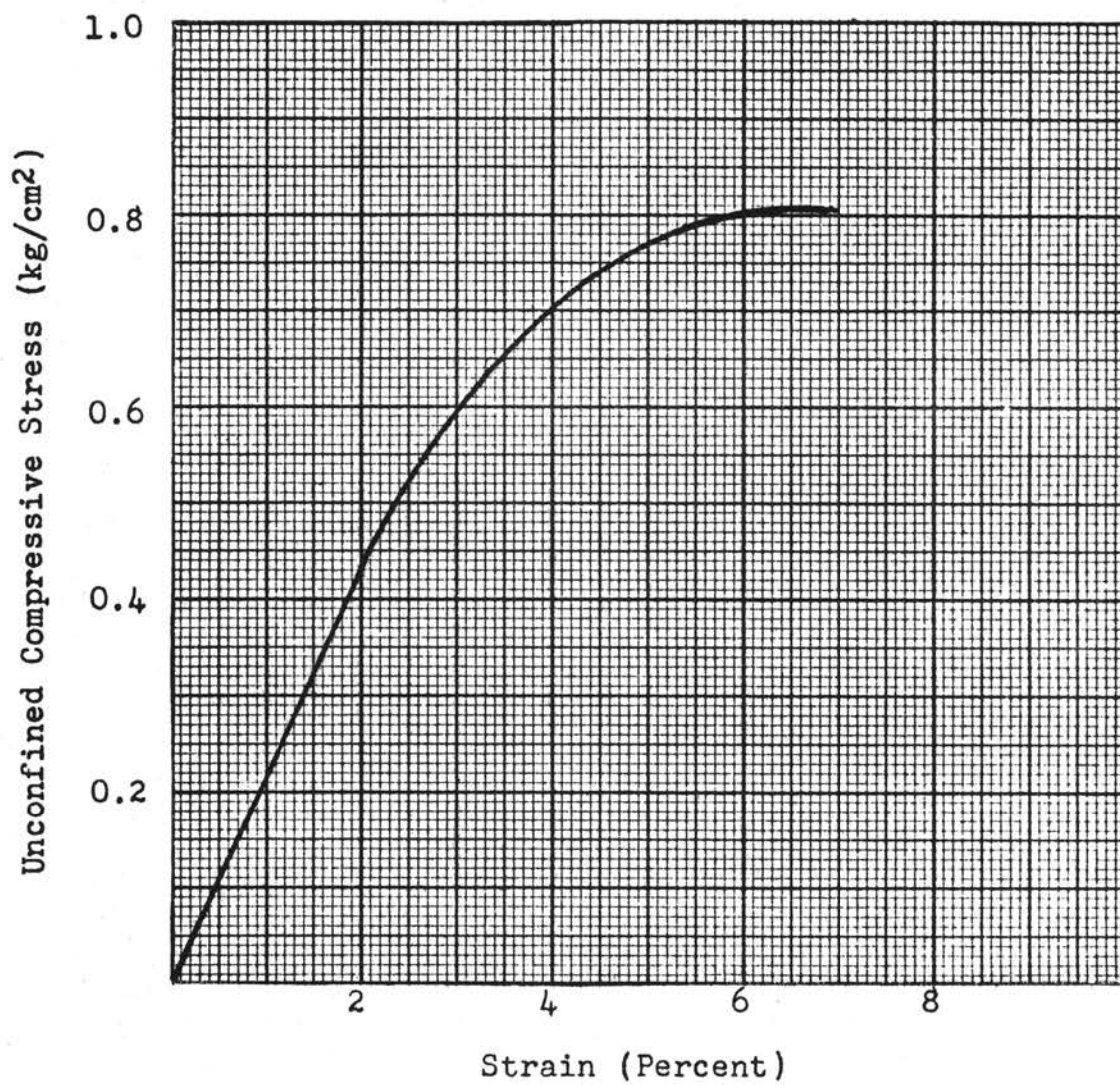


Figure 3

A Typical Stress-Strain Curve For
Terbec Rate of 0.20 Percent
(Group 1)

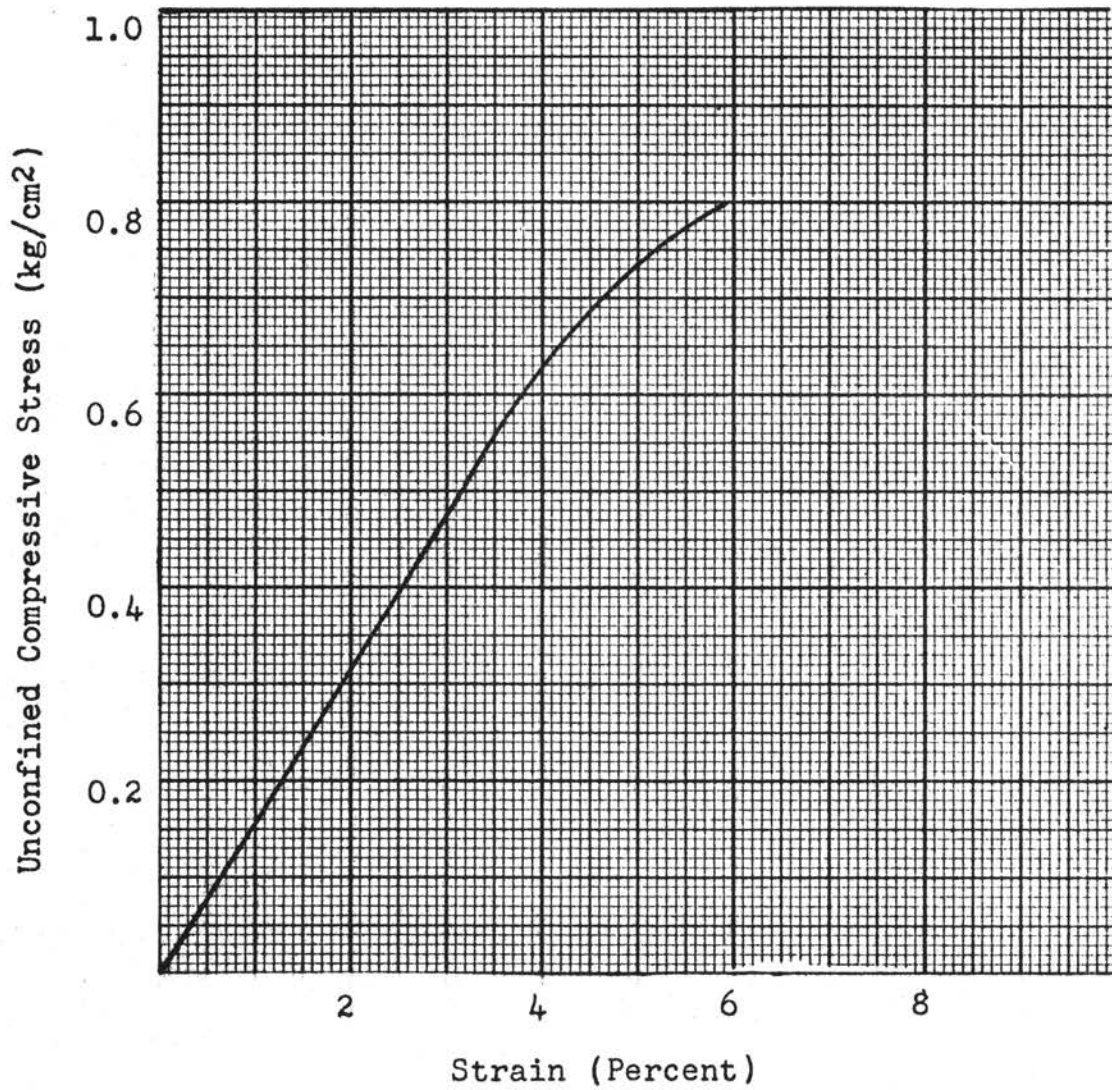


Figure 4

A Typical Stress-Strain Curve For
Terbec Rate of 0.20 Percent
(Group 2)

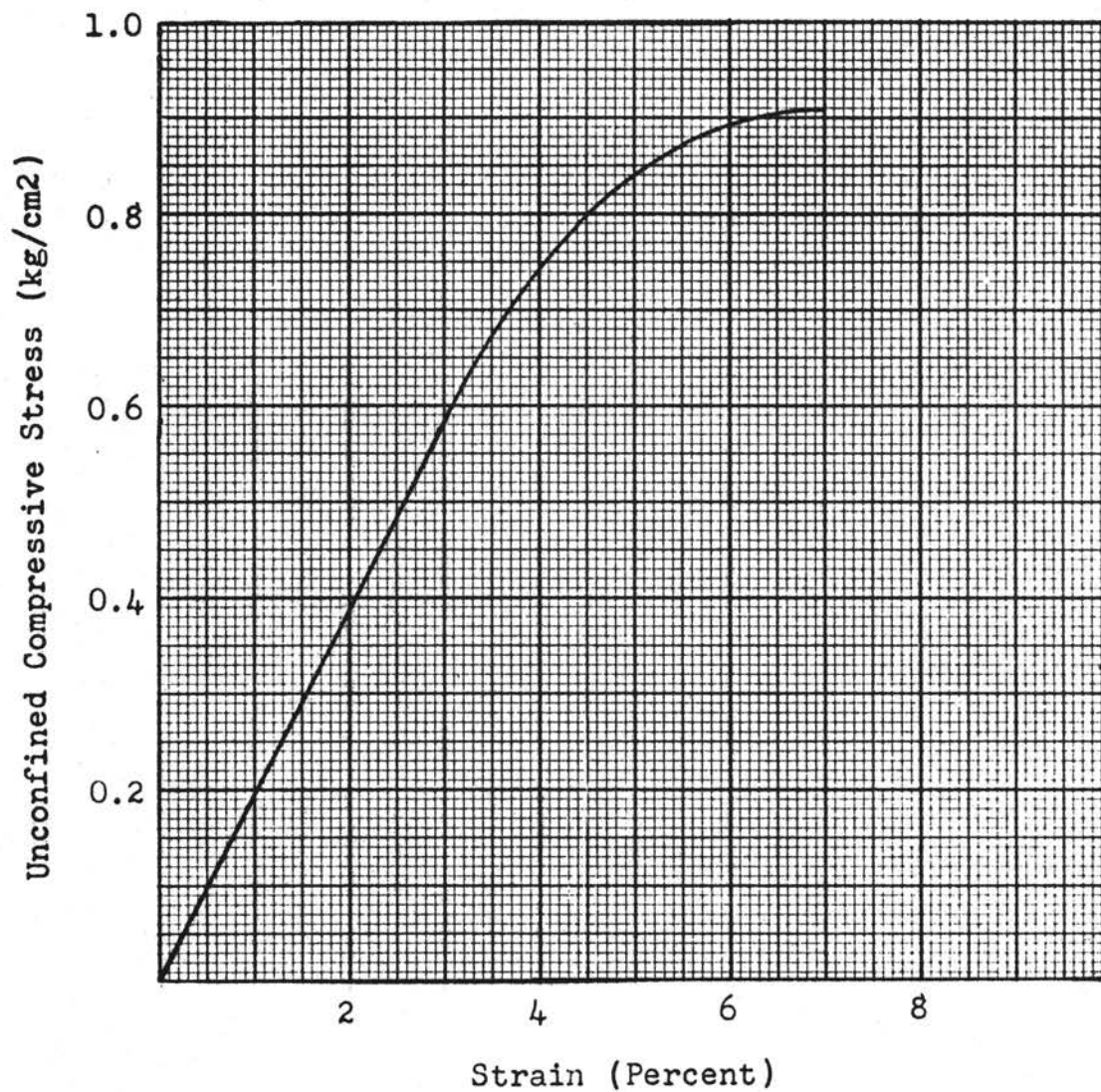


Figure 5
A Typical Stress-Strain Curve For
Terbec Rate of 0.25 Percent
(Group 1)

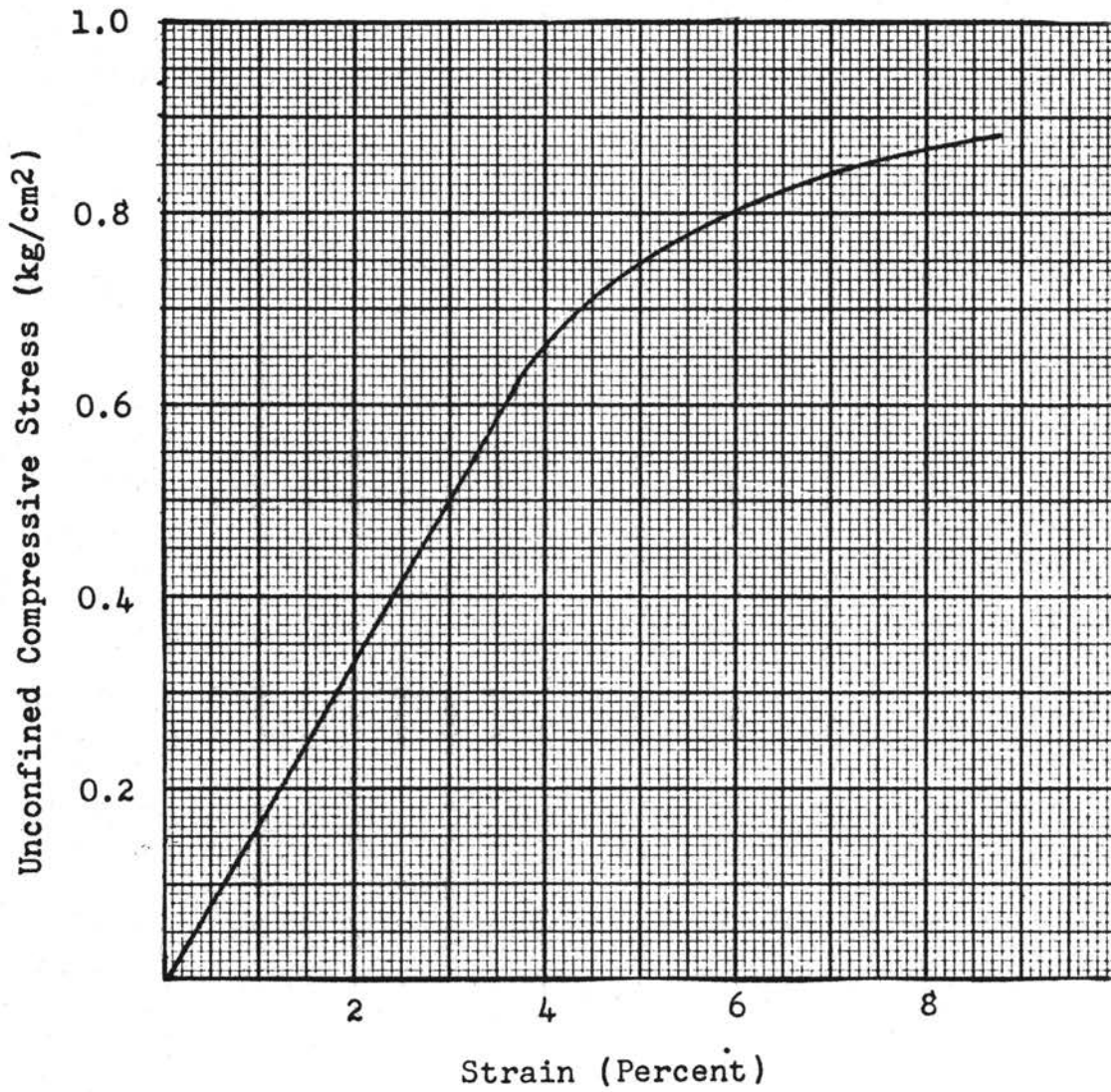


Figure 6

A Typical Stress-Strain Curve For

Terbec Rate of 0.25 Percent

(Group 2)

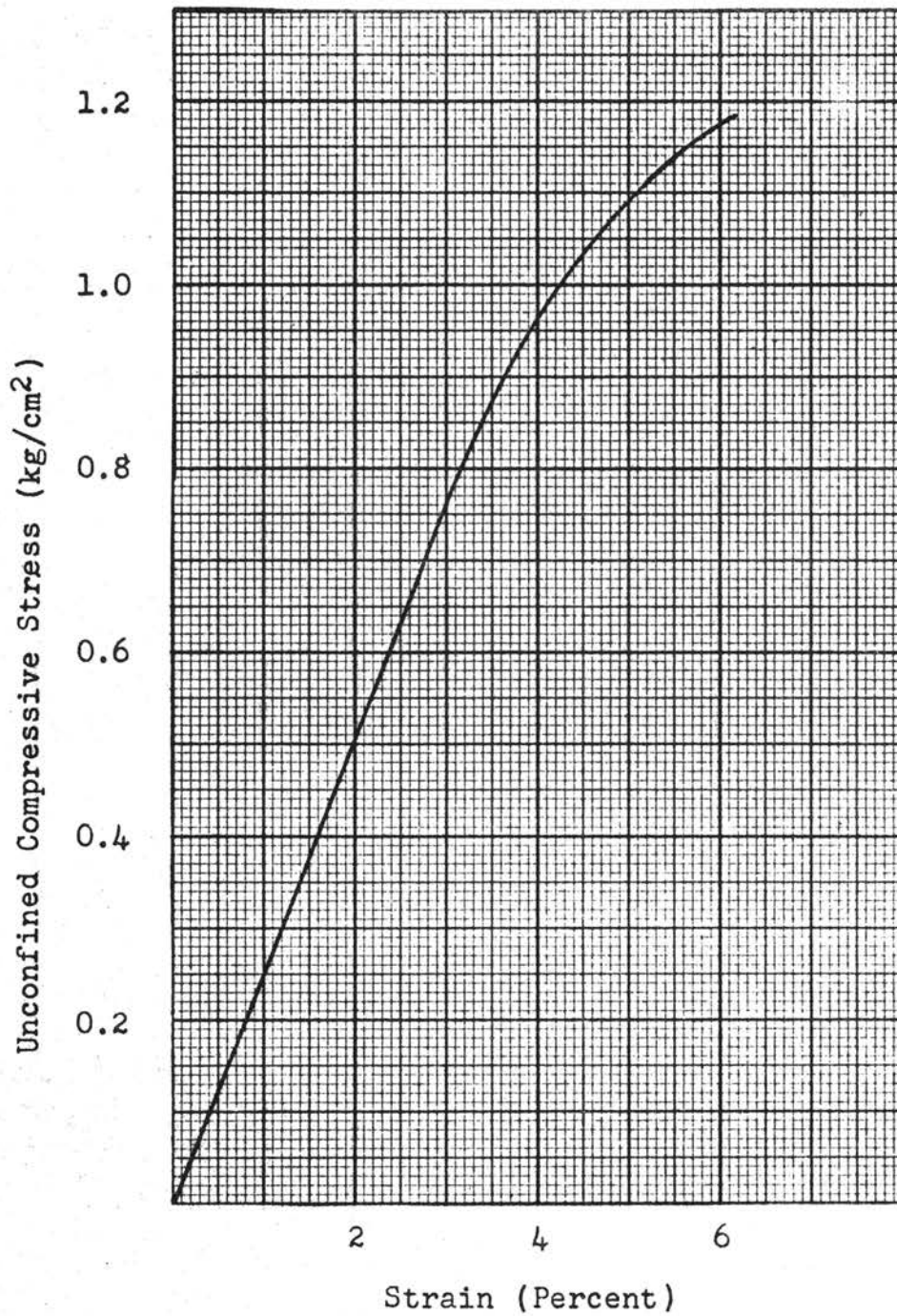


Figure 7

A Typical Stress-Strain Curve For

Terbec Rate of 0.50 Percent

(Group 1)

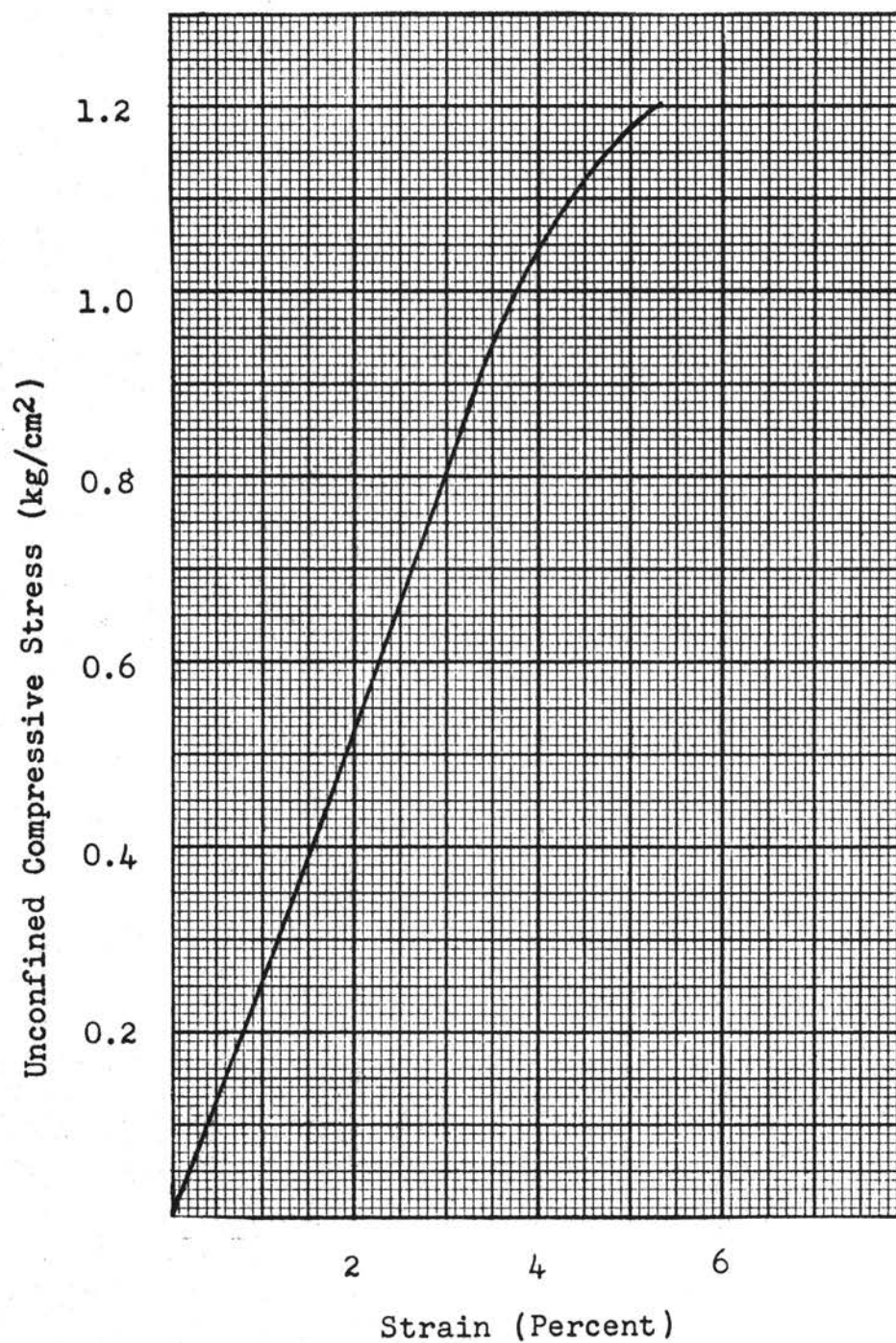


Figure 8

A Typical Stress-Strain Curve For
Terbec Rate of 0.50 Percent
(Group 2)

BIBLIOGRAPHY

- (1) Hemwall, J. B., D. T. Davidson, and H. H. Scott. "Stabilization of Soil with 4-Tert-Butylpyrocatechol." Chemical Soil Stabilization and Soil Aggregate Stabilization. Highway Research Board, Bulletin 357, 1962.
- (2) Grim, R. E. Clay Mineralogy. McGraw-Hill Book Co., Inc., York, Pa., pp. 1-10, 1953
- (3) Ibid. pp. 16-26.
- (4) Richards, C. W. Engineering Materials Science. Wadsworth Publishing Co., Inc., San Francisco, Calif., pp. 8-26, 1961.
- (5) Grim, R. E. Applied Clay Mineralogy. McGraw-Hill Book Co., Inc., York, Pa., pp. 7-16, 1962.
- (6) Means, R. E. and J. V. Parcher. Physical Properties of Soils. Charles E. Merrill Books, Inc., pp. 20-24, Columbus, Ohio, 1963.
- (7) Ibid. pp. 26-29.
- (8) Grim, R. E. Applied Clay Mineralogy. McGraw-Hill Book Co., Inc., York, Pa., pp. 16-24, 1962.
- (9) Ibid. pp. 29-34.
- (10) Winterkorn, J. F. "The Science of Soil Stabilization." Soil and Soil-Aggregate Stabilization. Highway Research Board, Bulletin No. 108, p. 1, 1955.
- (11) Murray, G. E. "Soil Stabilization by Chemical Means." Proceedings of the Conference on Soil Stabilization. Massachusetts Institute of Technology, p. 107, 1952.
- (12) Ibid. pp. 112-114.
- (13) Hemwall, J. B. "The Adsorption of 4-Tert-Bulylpro-catechol by Soil Clay Minerals." Proceedings of Internation Clay Conference, pp. 319-328, 1963.

- (14) Sherwood, P. T. "Soil Stabilization by the Use of Chemical Admixtures." Roads and Road Construction. pp. 102-110, April, 1961.
- (15) Lambe, T. W. "Soil Stabilization." Foundation Engineering. McGraw-Hill Book Co., Inc., York, Pa., pp. 351-437, 1962.
- (16) Matalucci, R. V. "Laboratory Experiments in the Stabilization of Clay with Gypsum." Master's Thesis. Oklahoma State University, 1963.
- (17) Bowman, J. W. "The Effects of Gypsum on the Strength of Clay." Master's Thesis. Oklahoma State University, 1963.
- (18) Mack, J. R. "Gypsum, Anhydrite and Plaster of Paris As Soil Stabilizers." Master's Thesis. Oklahoma State University, 1964.
- (19) Mitchell, M. L. "Sodium Hydroxide as a Permian Clay-Gypsum Stabilizer." Master's Thesis. Oklahoma State University, 1964.
- (20) Parcher, J. V. "Soil Stabilization Project Final Report." School of Civil Engineering Research Publication. Oklahoma State University, Stillwater, Oklahoma, No. R-22, 1964.

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