# SODIUM HYDROXIDE AS A PERMIAN CLAY-GYPSUM STABILIZER,

By

MAURICE LEROY MITCHELL Bachelor of Science Oklahoma State University Stillwater, Oklahoma

1963

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 May, 1964

© OKLAHOMA State University Library Jan 8 1935

Charles Block Frank States and States and States

## SODIUM HYDROXIDE AS A PERMIAN

CLAY-GYPSUM STABILIZER

Thesis Approved:

iser Thesis  $\cap$ Dean School 0

## ACKNOWLEDGMENT

The writer wishes to express sincere appreciation to his adviser, instructor, and supervisor, Professor J. V. Parcher, for his assistance throughout this investigation and for his thorough editing of this manuscript.

The writer also wishes to thank the Oklahoma State Highway Department and the United States Bureau of Public Roads for their financial support that enable him to continue graduate study and perform this investigation.

Appreciation is also acknowledged to Messrs. T. J. Chung, E. W. LeFevre, and P. C. Liu who shared the laboratory work for this investigation with the writer.

Also special appreciation is extended to P. C. Liu for his excellent preparation of many of the figures that were used in this manuscript.

Finally sincere appreciation is expressed to his parents, Mr. and Mrs. Claude A. Mitchell, for their assistance and encouragement in helping the writer to continue his college education.

iii

## TABLE OF CONTENTS

Chapte:	r	÷																		Ρ	age
I.	INŢRO	DUCTI	ON.	۰	• •	ρ	•	•	•	•	a	•	•	۰	•	•	•	•	٠	a	1
		Gener Origi Inves	al. n . tiga	.ti	on.	0 # 0	0 • 9	• •	•	•	•	a • •	•	•	• 9	4 4 •	• •	0	• •	9 4 6	1 2 4
II.	SOIL	STABI	LIZA	TI	ON	THE	lof	Υ	•	a	•	0	o	q	٥	o	. •	•	¢	.α	6
		Gener Funda Struc Ion E Clay- Stabi	al. ment ture xcha Wate liza	al ng r iti	Co f C e . Rel on	nce lay ati Rea	or or	is lin sh	ier ip	al s	• • • •	9 9 9 9 9	a 9 9 0	0 0 0 0 0	9 0 0 0 0 0	• • • • •	• • •	• • •	9 9 9 9 9 9	0 9 9 9 9	6 8 11 15 17 21
III.	MATER	IALS	USED	) : I.	NI	INVE	ISI	ΊG	AT	IO	N	•	•	•	٠	•	•	ą	٥	ę	23
		Permi Gypsu Loess Trace Sand	an R m Che	ed • •	Cl cal	ay .s.	• • • •	• • • •	•	•	• • •	0 0 0 0	e • •	9 9 9 9	• • •	•	• • •	• •	•	• • •	23 24 26 27 27
IV.	LABOR	ATORY	TES	TI	NG	PRC	CE	DU	RE	S	•	•	•	•	•	•	•	.•	•	٠	28
•		Gener Propo Uncon P V. C.	al. rtic fine Swe	ni: d	ng Com Te	and ipre	l M ess	ix io	in n	g Te	of st	M S	lat Spe	.er eci	ia me	ls ns	•	• • •	• • •	• • •	28 29 30 32
V.	TESTS	AND	RESU	ΓĹ	s.	e	<b>,</b> •	۰	•	•	•	•	•	٠	•	٠	•	٠	•	•	34
		Pilot Inter Sodiu Calic Loess Fine Swell	Stu medi m Hy um H -Gyps Gyps	idi at dr lyd su Pr	es. oxi rox st	tud de ide tud udi	ie St lie s	sud tu	ie di	• 5 5 • 5 • •	•	• • •	0	* * *	• • • •	• • •	• • •	• • • •	•	• • • •	34 35 37 41 42

# Chapter

VI.	CONCLUSIONS	AND	AND RECOMMENDATIONS FOR FUTU						URE	E				
	RESEARCH	• •	••	• •	• •	• •	• •	• •	٠	•	• •	•	47	
	Conclu	sion	<sup>3</sup>	• •	•_•	•••	•••	• •	•	•	•••	•	47	
	Recomm	enda	LION	s ror	r Fut	ure	Kes	earc.	n,	•	• •	•	49	
	FIGURES	• •	• •	• •	• •	••	• •	• •	•	•	• •	•	50	
	BIBLIOGRAPH	Y	•••	• •	• •	•••	•••	••	•	•	• •	•	86	

Page

# LIST OF FIGURES

Figure	Pa	ge
1.	Grain Size Distribution Curve for Permian Red Clay	50
2.	Grain Size Distribution Curve for Gypsum	51
3.	Grain Size Distribution Curve for Loess	52
4.	Effect of Various Trace Additives on the Strength of PRC-Gypsum Mixes	53
5.	Effect of Selected Trace Additives on the Strength of PRC-Gypsum Mixes	54
6.	Effect of Selected Trace Additives on the Strength of Permian Red Clay	55
7.	Effect of Sodium Hydroxide on the Unconfined Compressive Strength of Permian Clay	56
8.	Effect of Sodium Hydroxide on the Strength of PRC-Gypsum(5%)-NaOH Optimum Series	57
9.	Effect of Sodium Hydroxide on the Strength of PRC-Gypsum(10%)-NaOH Optimum Series	58
10.	Effect of Sodium Hydroxide on the Strength of PRC-Gypsum(20%)-NaOH Optimum Series	59
11.	Effect of Sodium Hydroxide on the Strength of PRC-Gypsum(30%)-NaOH Optimum Series	60
12.	Effect of Gypsum on the Strength of Mixtures of Permian Clay and 1% Sodium Hydroxide	61
13.	Effect of Gypsum on the Strength of Mixtures of Permian Clay and 2% Sodium Hydroxide	62
14.	Effect of Gypsum on the Strength of Mixtures of Permian Clay and 3% Sodium Hydroxide	63
15.	Effect of Gypsum on the Strength of Mixtures of Permian Clay and 5% Sodium Hydroxide	64

# Figure

16.	Effect of Moisture Content on the Strength of Permian Clay with 2% Sodium Hydroxide 6	55
17.	Effect of Moisture Content on the Strength of PRC-Gypsum(5%)-NaOH(2%) 6	6
18.	Effect of Moisture Content on the Strength of PRC-Gypsum(10%)-NaOH(2%) 6	57
19.	Effect of Moisture Content on the Strength of PRC-Gypsum(20%)-NaOH(2%) 6	8
20.	Effect of Moisture Content on the Strength of PRC-Gypsum(30%)-NaOH(2%) 6	9
21.	Effect of Moisture Content on the Strength of PRC-Gypsum(20%)-NaOH(5%)	0
22.	Effect of Sodium Hydroxide on the Dry Densities of Mixtures of Permian Clay and Gypsum 7	ŗı
23.	Effect of Lime on Strength of Permian Clay- Gypsum(20%)-Lime	2
24.	Effect of Various Admixtures on the Unconfined Compressive Strength of Permian Clay-Cured 90 Days	'3
25.	Effect of Various Admixtures on the Unconfined Compressive Strength of Permian Clay-Cured 28 Days	14
26.	Effect of Various Admixtures on the Unconfined Compressive Strength of Permian Clay-Cured 7 Days	15
27.	Effect of Curing Time on the Strength of Various Loess Combinations	6
28.	Effect of Fine Gypsum on the Unconfined Compressive Strength of Permian Clay 7	7
29.	Swelling Pressures of Mixtures of Permian Clay and Gypsum	8
30.	Swelling Pressures of Mixtures of Permian Clay and Quartz Sand	9
31.	Swelling Pressures of Mixtures of Permian Clay	20

Page

# Figure

32.	Effect of Sodium Hydroxide on the Swelling Pressure of Permian Clay	81
33.	Swelling Pressure of Mixtures of Permian Clay and 10% Gypsum with Sodium Hydroxide,	82
34.	Swelling Pressure of Mixtures of Permian Clay and 30% Gypsum with Sodium Hydroxide	83
35.	Swelling Pressure of Mixtures of Permian Clay and Gypsum with 2% Calcium Hydroxide	84
36.	Swelling Pressure of Mixtures of Permian Clay and Gypsum with 5% Calcium Hydroxide,	85

Page

## CHAPTER I

#### INTRODUCTION

#### General

During the present era of increasing volume of traffic and larger wheel loads of trucks and aircraft, the engineer is faced with the problem of building highways and airfields to take care of stresses of increased magnitude and frequency. The required design usually consists of a subgradebase-pavement combination that needs to be prepared from strong and durable materials. One of the problems here facing the engineer is that in many areas of the United States the supplies of suitable road-building materials are rapidly dwindling or have never existed. Therefore, the engineer must consider an economical means of improving the poorer quality materials which usually is the method of soil stabilization.

Soil stabilization may be defined as, "The process by which the physical properties of soil and soil-aggregate mixtures may be improved or altered by either mechanical treatment or with the addition of chemicals" (1). Mechanical stabilization is a process in which soil is combined with coarse and fine aggregates to produce a mixture which is superior to any of its components in strength and durability

l

characteristics. Chemical stabilization is accomplished by the addition of chemicals to modify or improve certain physical properties of the soils, such as the load-bearing capacity or strength of soils under various moisture conditions.

Chemical stabilization, which is to be studied here, has made many worthwhile advancements during the past few years in the use of bituminous products, portland cement and lime compounds as very effective stabilizers for many soils and soil conditions. From a consideration of the investigations that have been conducted on the use of the above compounds and many other chemicals, no one or two chemicals have been found that will remedy or stabilize every soil condition that the engineer becomes concerned with. Since some chemicals when added to the soil will be more destructive than constructive, the engineer must be careful to use chemicals that will impart to the soil the properties he is interested in obtaining.

## Origin

One of the troublesome soil types involved in many areas is the fine-grained clay which, due to its particle size and mineral composition, imparts properties of plasticity, low strength, shrinkage, and swelling to the soil. These properties are detrimental if they are associated with the soil which is to be used in the construction of subgrades and bases of the highways required by today's traffic.

The plastic red clays of the Permian Red Beds that exist in much of Oklahoma, especially in the western and central counties, are very widespread and are a constant source of trouble to the engineers who try to build upon them. The strength and volume stability of the Permian clays are quite susceptible to changes in moisture conditions. Due to the cyclic effect of moist and dry seasons in Oklahoma, the water content of the clay varies considerably causing volume instability which is detrimental to anything built with or upon these clays.

Due to the increasing scarcity of top quality road building materials in Western Oklahoma and because there are abundant deposits of gypsum, the gypsum is being considered as a material to be used as a stabilizer for the Permian Red clay. Very little literature (2-6) is available about the use of gypsum as a soil stabilizer but what is available indicates a possibility that gypsum might be acceptable.

Some recent research at Oklahoma State University by Matalucci(4), Bowman(5), and Mack(6) concerning the stabilization of these Permian Red clays with gypsum indicates that gypsum by itself does not have much effect on the unconfined strength but does reduce the swelling, reduces the liquid limit, and improves the workability of the Permian Red clay.

From these indications and results, research was conducted at Oklahoma State University, sponsored by the Bureau

of Public Roads and the Oklahoma State Highway Department to determine if a chemical compound in small quanities could be added to a mixture of Permian Red clay and gypsum that would provide an increase in the unconfined strength and further reduction in the swelling characteristics of the clay soil.

## Investigation

This investigation was concerned with the search for chemicals that could be used as additives for the purpose of stabilizing mixtures of Permian Red clay and gypsum. The chemical additive sought was one that would impart to the Permian clay and gypsum mixture, increased shear strength and volume stability.

The investigation was initiated with a study of seventeen trace chemicals added to Permian Red clay and gypsum mixtures. Based upon the results obtained from unconfined compressive strength tests of clay-gypsum mixtures using these trace chemicals; the three that showed greatest strength increases over those clay-gypsum mixtures without trace chemicals were selected for additional study. These three were reduced to one, sodium hydroxide (NaOH), with the completion of further strength tests.

The bulk of the investigation was then concerned with the strength and swell of Permian Red clay and gypsum mixtures using sodium hydroxide as the trace chemical. Tests using lime,  $Ca(OH)_2$ , were also conducted as a reference to

the effectiveness of sodium hydroxide. Lime was used since considerable research has been performed and published, concerning its strength and volume change characteristics when used with many different soil types.

The shear strengths of the test specimens were measured by an unconfined compression test. The unconfined compression test was chosen because a simple and easily performed test was considered a very valuable asset, due to the large number of specimens that were to be tested.

The unconfined test specimens were molded by the use of the Harvard Miniature Compaction Device because of the relatively small amount of material required and the ease of handling, storing and testing.

The swelling potential of the soil specimens was determined by the use of the Potential Volume Change (PVC) Meter. The PVC meter was selected for its ease of operation and for its characteristic of measuring one dimensional swell quickly and effectively which was deemed desirable.

## CHAPTER II

### SOIL STABILIZATION THEORY

## General

A problem which continually confronts the engineer is the one dealing with procedures and techniques by which otherwise unsuitable soils may be improved by stabilization for engineering purposes. Soil stabilization in its widest meaning (Winterkorn 7), encompasses every physical, physicochemical and chemical method developed and used to make a soil perform better its desired engineering purpose. In its specific meaning as commonly understood in highway and airfield engineering, soil stabilization is the name given to those methods of construction in which unsuited soils are treated to provide subbase and base courses which can carry the applied traffic loads under all normal conditions of moisture and traffic for an economic service period.

Methods of stabilizing soils as stated by Murray (8), can be divided into two classes--physical and chemical. The methods of applying the physical and chemical treatments are many and widely varied. Physical or mechanical stabilization is not only the earliest form of designed and controlled stabilization, but the ideas and procedures used in obtaining this mechanical stabilization are frequently common

to those used in other types of stabilization. Physical methods of stabilization do not, however, alter the inherent nature of the soil. Chemical methods of stabilization do alter the inherent properties of soils and it is this ability that gives chemical stabilization its great promise.

For the purpose of this investigation, soil stabilization will be defined as the process by which the physical properties of soil may be improved or altered by either mechanical treatment or with the addition of chemicals (Woods 1). The process by which the strength and the durability characteristics of soil and soil aggregate mixtures are increased by the utilization of the proper combination of soil with coarse and fine aggregate, is mechanical stabilization.

Chemical stabilization (Wooltorton 9) consists mainly of adding to a soil material an optimum percentage of a material of opposite ion polarity to produce by exchange phenomena, a mixture of greater strength or a mixture in which the clay mineral particles or aggregates are surrounded by a shield resistant to moisture penetration. This problem is essentially one of a surface-chemical type. Not only can the usual soil properties be altered, but also entirely new soil properties such as tensile strength and flexibility can be produced in soil by the proper chemical treatment.

The problem of stabilizing soil, as conceived by Hauser (10), becomes particularly important when one deals with fine-grained clays, most of which contain montmorillonite.

In these clays, water will have a decided effect since these soils lose much of their shear strength when they become very wet. Most of the stabilization methods now in use encounter increasing difficulties as the size of the dominant soil particles decreases. Due to these difficulties the investigation for better stabilizers for the finegrained soils is being pursued quite actively.

Clays, in soil stabilization, must always be considered as very important and often major soil components; therefore, a basic knowledge of their structure, composition, and morphology is of great importance.

Despite the emphasis placed here on the clay minerals and their properties, it should not be forgotten that clay minerals in natural soils frequently have adsorbed on their surfaces organic matter resulting from the decomposition of plants or animals, thereby becoming contaminated. Because of this, surface activity of these clays will differ considerably from that of pure clay.

### Fundamental Concepts

Murray (8) states in his article that every mass is united by a three-dimensional network of forces. These forces are considered to be of two major types--chemical and physical. Chemical forces are those forces that exist between the atoms. Physical forces are those that exist between the molecules and discrete aggregates and are essentially surface forces. Here a molecule is considered to be a group of atoms united by chemical forces.

The two chemical forces that are of major importance in soil stabilization are ionic bond and covalent bond. Since these bonds exist between atoms, considerable energy is required to break the atoms apart, and once broken apart, they do not readily combine again. The ionic bond is formed by a transfer of electrons from one atom to another to form charged particles. These atoms are than held together by the electrostatic forces developed due to these charged particles or ions. These bonds are the simplest type, extremely strong, and considerable energy is required to break the atoms apart.

The covalent bond is formed by a sharing of electrons by the atoms. This sharing of electrons is accomplished by the transfer of energy between the different atoms as the electrons spin about their nucleuses in opposite directions. These bonds are less strong then the ionic and exist generally in non-electrolytes and between elements that are electronegative.

There also exists another chemical force of minor importance known as the hydrogen bond. It is a bond caused by the attraction of two atoms or anions to the hydrogen atom's single proton. This develops a weak bond because normally when only one anion is bonded to each proton a stronger bond arises.

The physical forces that are of interest are the Van der Waals and polar forces. These forces generally are

considerably weaker than the chemical forces and can be broken quite easily by physical methods such as heat and solvents. Usually upon the removal of the disturbing effect, the material reverts to its original unaltered form,

Many molecules have been shown to have an uneven distribution of electrons within the molecule caused by a lack of symmetry, thereby producing localized electrical charges. These types of molecules are termed dipoles. The Van der Waals forces exist between those molecules that are not dipoles whereas the polar forces exist between those molecules that do have localized electrical charges.

In a mass of soil, the interatom forces are the chemical or primary forces whereas the aggregation of the particles is due to the physical or secondary forces. In many types of soils these secondary forces can be of considerable magnitude because, since they are surface forces, they depend upon the amount and type of surface present.

In the ultimate analysis stabilization, or more generally, modification of the properties of a soil is effected by altering the forces that unite the individual soil particles. This may be accomplished by altering the secondary forces already present in the soil or by introducing a new material that acts through its own primary or secondary forces.

## Structure of Clay Minerals

Hauser (10) states that in 1923, investigators were able to prove by the use of X-ray diffraction studies that clays were crystalline in structure. Since then our knowledge of the structure of clays has greatly increased and it has been found that the common clay minerals are all composed of hydrous aluminum-silicates, which have very weak basal cleavage planes that allow them to be broken into extremely thin sheets (Means and Parcher 11).

These hydrous aluminum silicate minerals, as dicussed by Means and Parcher (11), are composed generally of two fundamental building blocks, which are a tetrahedral silica unit and an octahedral hydrous aluminum oxide unit.

The tetrahedral silica unit is a tetrahedral arrangement of four oxygen ions with a silicon ion enclosed within the oxygen ion arrangement. The radii and distances between the silicon and oxygen ions are such that they permit the four oxygens to touch, leaving a space just large enough to include the silicon ion. These tetrahedral silica units become bonded together with other silica units by sharing of each of the oxygen ions in the base with another tetrahedral silica unit. The strong horizontal forces that are developed between these units with exchanged and shared electrons are by ionic or covalent bonds. Due to the strong bond provided by the sharing of the oxygen ions with two molecules, a sheet structure of silicon ions between two layers of oxygen ions is formed. In this arrangement the

oxygen bases of the tetrahedrals are in a common plane with the apexes all oriented in one direction. The net charge of the unit becomes a minus one, because of the minus five produced by the shared oxygen ions and only a plus four from the silicon ions.

The octahedral hydrous aluminum oxide unit (gibbsite) is an octahedral arrangement of six hydroxyl ions enclosing an aluminum ion. These octahedral units are bound together in a sheet structure with each hydroxyl ion common to three other octahedral units. Because aluminum has a plus three charge and the sharing of hydroxyl ions contributes only a minus two charge there results a net charge of the octahedral unit of plus one.

Under some special conditions, the aluminum ion may be substitued for an iron or magnesium ion without changing the crystalline form. According to Hauser (10), there is induced into the structure of these units with substituted ions, strains caused by the magnesium ion being larger than the aluminum ion. The strain is developed by the magnesium ion having to stretch the aluminum cavity to be able to fit itself into the cavity. Such a substitution of one element for another in the formation of a crystal without changing the crystalline form is known as isomorphous substitution.

The different clay minerals that exist are formed by bonding together different combinations of the molecular sheets of silica and gibbsite. When the apex's of the silica units become intergrown with the hydroxyl ions of

the gibbsite units, a two layer elemental sheet is formed with a strong ionic bond because of the opposite charges of the two units.

If in the formation of the combined silica-gibbsite sheet, there are more silica units present than aluminumhydroxyl units then a silica-gibbsite sheet with a surplus of negative charges is formed. This combined sheet has the shared oxygen ions of the silica sheet exposed on one side and the shared hydroxyl ions of the gibbsite sheet exposed on the other.

In the formation of minerals, sometimes there becomes an excess of silica ions which causes another elemental sheet to be formed by the addition of another silica sheet being bonded to the other side of the gibbsite sheet. This new silica-gibbsite-silica sheet structure becomes even more negative if magnesium replaces the aluminum by isomorphic substitution. The negatively charged surfaces then may attract, and be held apart by, dipolar water molecules when their positive ends are oriented toward the negative surfaces of the elemental sheets. The negative surfaces of these elemental sheets may also be held together with cations of potassium, calcium, sodium and some more of the common elements.

The distance between these elemental sheets is controlled by the amount of dipolar water available for reaction with the negative surfaces.

The three clay minerals montmorillonite, illite and

kaolinite, are the principal minerals of clay and the cause of many of the clay's characteristic properties.

Montmorillonite is a hydrous aluminum silicate crystal formed by layers of silica-gibbsite-silica sheets separated by attracted water layers between the negatively charged silica sheets. A very important characteristic of the montmorillonite clay is that it has an expanding lattice. This expanding lattice is caused by the varying amounts of water that is available and can be attracted between the negatively charged silica sheets. This expanding lattice is developed by the power of the silica-gibbsite-silica sheet being able to attract a water thickness up to twenty times the thickness of the elemental sheet.

Illite is a hydrous aluminum silicate crystal that is similiar to montmorillonite except that the adjacent silica layers are bonded together with potassium ions instead of water. Because conthe cation bond of the illite is stronger than the water bond of the montmorillonite the tendency of the illite lattice to expand is not as great as that of the montmorillonite.

Kaolinite is also a hydrous aluminum silicate crystal but is is formed by a stacking of alternate layers of silicate and gibbsite sheets bonded together by a hydrogen bond. Because this hydrogen bond between the base surfaces of the silica layer and the gibbsite layer is quite strong the lattice is fairly stable and does not expand readily. Also because of this relatively strong hydrogen bond the

particles of kaolinite do not break down into single silicagibbsite sheets but clusters of sheets with high negative charges which can attract very thick layers of water.

## Ion Exchange

Ion exchange and the exchange reactions are important properties of clay minerals that must also be considered in the study of clay soils (Grim 12). These properties of adsorbing and retaining certain anions and cations in an exchangeable state and then exchanging them by treatment with other ions in a water solution are of extreme importance in all fields of the study of clay materials. Grim (13) states that these exchangeable ions are held around the outside of the silica-aluminum clay mineral structural unit and that the exchange reaction usually will not affect the structure of the silica-aluminum crystal. Ion exchange and reactions are of two major types -- cation exchange and anion exchange. Cation exchange is considered to be caused in clay minerals by one of three types of mechanisms. One method is that the broken bonds along the noncleavage surfaces of the silica-aluminum units leave unsatisfied charges that can be balanced by the adsorption of cations. Because the exchange capacity increases as the number of broken bonds increase and these bonds increase as the particle size decreases, this method becomes the major cause of cation exchange in kaolinite and illite minerals.

A second method is by substitution within the lattice

of the trivalent aluminum for ions of lower valence, usually magnesium. This type of substitution accounts for approximately eighty percent of the cation-exchange capacity of the montmorillonite minerals.

A third method is by the replacement of the hydrogen of the exposed hydroxyls with an exchangeable cation. This method is of importance in the cation exchange of kaolinite but there is some doubt as to how strong a factor it is because normally these exposed hydroxyls are present along the broken edges of the clay minerals and the first method probably has more effect.

The rate of cation-exchange reactions varies with the clay mineral, with the concentration of the cations, and with the nature and concentration of the anions. These reactions are rapid for kaolinite, slower for montmorillonite and extremely slow for illitic minerals.

Anion exchange reactions are extremely difficult to investigate because the clay minerals will decompose during the course of the reactions. It is believed that anion exchange can take place by two major methods. First by the reaction of anions with the broken bonds around the edges of the clay mineral particles and second by a reaction with the hydroxyls on the surfaces of the clay mineral particles.

The exchange capacity of materials is measured and expressed in terms of milliequivalents per 100 grams at a pH\_ of seven.

## Clay-Water Relationships

One of the important physical properties of soils with which the engineer is concerned is volume change. In clays, the volume change can be of considerable magnitude and is usually developed by a change in moisture content causing either a shrinkage or swelling process. Therefore, the engineer becomes interested in the amount and types of moisture present and the effects of them upon the clay structures.

The water that is attracted and held by the clay material is of four different types (Grim 14). The first one is not of much concern because it is affected only by extremely high temperatures; but the last three occur at normal temperatures and are classed according to the water's relation to the clay mineral components and texture.

- (1) Lattice water--this is not considered to be ordinary water because it is composed of the hydroxyl groups of the crystalline structure.
- (2) Pore water--this water has all of the properties of ordinary water and is the water in the pores, on the surfaces and around the edges of the particles composing the clay material.
- (3) Adsorded water--this is the non-liquid water contained on the surfaces of the minerals, both the exterior and the interior surfaces on the minerals with expanding lattices. This water is highly immobile and strongly oriented because of the

dipole characteristics of the water molecules and the normally negatively charged surfaces of the clay mineral particles. This water is solid or non-liquid and forms relatively thin layers varying from one to ten molecules thick.

(4) Attached water--this is the layers of ordinary water contained around the adsorbed water. It's viscosity varies from that of thick molasses next to the adsorbed water around the particles to that of ordinary water at varying distances from the particles.

The volume change behavior of soils is extremely influenced by the amount and type of clay mineral present along with the changes in moisture condition. Moisture conditions is a general term relating to the physical environment of the soil which determines the movement of water through the soil.

Because of the electrical, colloidal nature of clay, the clay particles have a great attraction for ions and water in the double-layer association of water about the particles (Lambe 15).

An equation of statics may be written that will predict volume change and relate the intra-particle stresses with the engineers applied and measured stresses.

The equation is:

F= +- 1 = R-A

where:

- ➡ the effective or intergranular stress which is the force transmitted between interacting particles per unit of soil and can be well correlated with soil behavior.
- + = total external stress applied to the soil.
- *M* = pressure in the free pore water of the soil mass which is that water outside the influence of the physico-chemical forces from the particles.
  - $R^{=}$  the repulsive pressure which arises from the electrical nature of the particles.
    - A = the attractive pressure between clay particles which also originates from the electrical nature of the particles.

This equation is interpreted to mean that for a given soil-water system with a given interparticle spacing, there is a repulsive pressure between the soil particles (R-A). It requires the application of an effective stress (4-24) to the soil to maintain volume equilibrium.

Therefore, any procedure that changes the effective stress on a given soil-water system will cause a volume change. The effective stress can be changed either by changes in  $\bigstar$ , the externally applied load, or by changes in the pore pressure  $\mu$ . Changes in the pore pressure are caused by changes in the moisture conditions of the soil such as wetting and drying and are usually referred to as swelling and shrinkage, respectively. Although volume change due to swelling and shrinkage are studied in terms of changes in effective stress, it should be understood that the particle characteristics of the soil determine the amount of volume change for a given effective stress change. One of these characteristics is that as the soil particles become smaller, the surface area of the clay increases. This greater surface area requires a greater volume change per change in particle spacing to make delta (R-A) equal to delta  $\mathbf{F}$ .

Soil shrinkage, from an effective stress viewpoint, is caused by the evaporation of water from the exterior surfaces, thereby building up a tension in the pore water. This negative pore water stress increases the effective stress which leads to a reduction in the interparticle spacing. This reduced spacing causes water to flow from between the particles to the surface where it evaporates. The reduction of the particle spacing along with the loss of water causes the soil to shrink in volume.

Factors that affect shrinkage are the initial water content, the degree of saturation, the drying conditions and the size of the sample.

Soil swelling is usually due to a decrease in the effective stress acting on the soil mass so that the repulsive pressure (R-A) created between the interacting soil particles pushes the particles apart, thereby causing swelling of the soil mass.

Because of the double-layer deficiency of water in

partially saturated soils, pore water tensions are developed which will cause water to flow into the soil whenever water becomes available. The water will continue to flow into the soil forcing the double layers to expand and forcing the clay particles apart, until the repulsive pressures between the particles become equal to the applied effective stress.

Some important factors that effect the swelling behavior of soils are:

- (1) composition of the soil,
- (2) initial water content and dry density,
- (3) arrangement and orientation of the soil particles,
- (4) chemical properties of the pore fluid,
- (5) confining pressure applied during swelling, and
- (6) time allowed for swelling.

## Stabilization Reactions

After a stabilizing agent is added to a soil, some type of reaction must take place to complete the stabilization. (Murray 16). These reactions may be either physical or chemical reactions.

A physical reaction is one in which the stabilizing agent is created before it is added to the soil, but requires an alteration to become effective. The most important physical reactions are those reactions caused by change in temperature, hydration, evaporation and absorption, An example of an physical reaction is that of adding portland cement to the soil, which then becomes stabilized by the alteration of the non-hydrated cement into its hydrated form.

A chemical reaction is one that causes a new material to be formed with different properties than the original material. The major types of chemical reactions that seem to be of importance are:

- (1) ion exchange--the replacing of ions attached to the soil particles by a different ion,
- (2) precipitation--formation of an insoluble compound which precipitates out of solution because of the reactions of other chemicals in solution,
  - (3) polymerization--the creation of large compounds or molecules from the reaction of simple compounds,
  - (4) oxidation--methods involving the use of oxidation techniques to produce an stabilizing agent.

## CHAPTER III

### MATERIALS USED IN INVESTIGATION

## Permian Red Clay

The clay material used was obtained from a naturally occuring deposit of clay associated with the Permian Red clay beds located throughout Oklahoma. The Permian Red clay was chosen because of its troublesome qualities to the highway engineer in construction and the large quanities located throughout many regions of Oklahoma.

The Permian Red clay used was excavated from a depth of two to five feet from the basement of the Life Sciences Building located on the Oklahoma State University campus at Stillwater, Oklahoma. The clay was broken into small particles and allowed to air dry to approximately three percent water content. Then the clay was pulverized and screened through a number ten sieve. Figure (1) shows the grain size distribution obtained from a hydrometer analysis of the soil. The soil was found to have a liquid limit of 40.5, a plastic limit of 15.0, and a plastic index of 25.5, using the standard Casagrande procedures. A standard AASHO compaction test produced a maximum dry density of 110 pcf, at an optimum water content of 17 percent. The specific gravity was determined to be 2.72. The base exchange capacity

of the Permian Red clay was found to be 13.0 M.E./100 grams, using both the Bower and the Kjeldahl methods. The Permian Red clay consists of approximately 40% montmorillonite, 30 to 35% illite, and 25 to 30% unidentified material. The unidentified material is believed to be quartz, kaolinite, and iron oxide.

#### Gypsum

The gypsum used was supplied by the United States Gypsum Company plant located at Southard, Oklahoma. The gypsum obtained was divided into three parts and then sieved through mechanical sieves to obtained gypsum finer than No. 10, No. 100, and No. 270. The grain size distribution curve for the gypsum is shown in figure (2). The specific gravity of the gypsum was found to be 2.32 and the hardness of the Mohs scale to be 2.0. The coefficient of uniformity was determined to be 5.0.

Because of the large quantities of gypsum located in Western Oklahoma, its economy in mining and its controllable properties, the gypsum is being considered as a stabilizer for fine-grained soils.

Gypsum  $(CaSO_4 \cdot H_2O)$  which is calcium sulfate with two molecules of combined water, is a useful industrial mineral found abundantly on the earth's surface (17). It is inexpensive to mine and process and its calcined products have a wide range of readily controllable properties such as strength, density and setting time. The valuable properties of gypsum result from its two molecules of combined water. After moderate heating, three fourths of this combined water is removed and gypsum becomes a plaster which will set with addition of water.

Man has long recognized the useful qualtities of gypsum. It was utilized by the Egyptians in the construction of the pyramids and later by the Romans for important structures. The use of gypsum continued through the Middle Ages and into modern times. Up until a few years ago gypsum was used mostly as a soil conditioner, but in recent decades it has become much more important as a major material for the building industry. Its major uses is in making plaster boards, making of Plaster of Paris, as a retarder in Portland Cement, and as a filler in paints and paper.

Gypsum is generally found as a sedimentary rock associated with limestone, dolomite, shale, or clay, in strata deposited from early Paleozoic to recent time. The Permian was particularly noteworthy for the development of enormous deposits, including the famous gypsum beds stretching from Kansas across Western Oklahoma and into Texas. In past geological times, notably during the arid climate of the Permian and Trias, the evaporation of seawater, intermittently replenished with further supplies, resulted in the accumulation of great thickness of gypsum saline residue deposits, which have become an increasingly important material in today's building and construction industry.

#### Loess

The loess material used was obtained from a naturally occuring deposit located in Grant County, Kansas, near Ulysses. The loess material was investigated to determine if gypsum and trace chemicals could be used as effective stabilizers.

Excavation of the soil ranged from a depth of five to seven feet in a test pit that had been excavated for foundation studies for the Cities Service Helium plant. The soil was taken out of the test pit in blocks about one foot square. The blocks were wrapped, waxed and stored in a humid room to preserve the soil in its natural condition awaiting time of its use.

The soil was a light-grey silty-clay loess material which contained noticable quantities of calicum and magnesium along with traces of sodium and potassium. These compounds existed probably in the form of salts deposited from solution by evaporation.

The soil was taken from its storage condition, broken into small particles by hand and allowed to air dry. Then the soil was sieved through a number ten size sieve and a series of property determination tests was conducted. Figure (3) shows the grain size distrubtion curve obtained from a hydrometer analysis. A liquid limit of 33.0, a plastic limit of 16.0 and plastic index of 17.0 was obtained by use of the standard Casagrande procedures. A standard AASHO compaction test yielded a maximum dry density of

99.0 pcf. and an optimum moisture content of 22.0%. The specific gravity was determined to be 2.69.

## Trace Chemicals

The chemicals used as trace chemicals were of a reagent type purchased from the chemistry storeroom. The chemistry storeroom is operated by personnel in the Chemistry department and is located in the Chemistry-Physics Building located on the Oklahoma State University campus.

#### Sand

The sand used in the PVC test was an Ottawa (quartz) sand purchased from the Dolese Company in Oklahoma City, Oklahoma.

## CHAPTER IV

## LABORATORY TESTING PROCEDURES

#### General

The condition of the soil is extremely dependent upon the amount of moisture it contains; therefore, caution must be exercised in the determination of its water content. The initial water content of all soils used was determined by dividing the dry weight of the materials into the loss in weight during drying.

These materials were dried in a drying oven maintained at 60 to  $65^{\circ}$  C. to drive off the initial or free moisture. Drying temperatures of greater than  $65^{\circ}$  C. could not be used with any mixtures containing gypsum. Gypsum will calcine at temperatures above  $65^{\circ}$  C., thereby driving off part of the water of crystallization, which greatly affects the amount of moisture present. The true moisture content was used to determine the dry density of the specimens and to check for undesirable moisture changes that might have occured during the test. The true moisture content was determined upon the portion of soil that was recovered after the specimen was tested. Dividing the dry weight of this soil into the loss in weight due to 24 hours of drying at  $65^{\circ}$  C. is considered the true water content.
Compaction of soil specimens is also dependent upon the amount of moisture present in the soil. This comes about from the fact that for a given compactive effort, a soil will obtain its maximum density at a particular moisture content. This moisture content is arrived at by plotting a curve for a series of specimens prepared at different moisture contents. The water content at the point of maximum density was considered to be the optimum moisture content. It was at these optimum conditions that almost all of the specimens were prepared.

Proportioning and Mixing of Materials

Proportioning of the materials to be stabilized was done while they were in an air-dried condition. The proportions are based on the total dry weight of the mixture. Such as, a mixture of 10% gypsum and 90% Permian Red clay means that 10% of the total dry weight is gypsum and 90% is clay. The dry materials were throughly mixed by hand until the appearance of the material was uniform.

The amount of distilled water required for the mix was based upon the total weight of the dry materials, while considering the initial water content. If 12% water content was desired for a 500 gram mix and the initial water content was 4% then ( $0.12-\pm0.04$ ) (500) or 40 grams of distilled water was added to the dry mix. The water was added gradually to the materials while they were being mixed thoroughly. The mixing was halted when the appearance of the materials developed a uniform texture.

The trace chemicals were added on the same basis as the water--a certain percent of the total dry weight. Some of the trace chemicals were added to the dry materials in a powdered form while others were dissolved in the distilled water that was to be used. When the chemicals were dissolved, the liquid solution in some cases had to be cooled back to room temperature because the solution became hot when the chemicals dissolved.

Unconfined Compression Test Specimens

Immediately upon completion of the mixing, the soil was compacted into cylindrical test specimens using the Harvard Miniature Compaction Device designed by S. D. Wilson. The compactive energy was obtained by the use of a handoperated spring loaded tamper. The tamper was calibrated to exert a twenty pound force per blow with a 0.375 inch diameter piston. The material was compacted in three equal layers of material with 25 blows per layer equally spread over the material. The mold in which the specimens were formed was 1.31 inches in diameter and 2.82 inches long. The correctlength of 2.82 inches was obtained by trimming the ends of the specimen with a straight edge. The specimens were then extruded from their molds by the use of a hand operated extruder, care being taken not to disturb the specimens, and weighed to determine their wet density. Because the mold volume is 1 / 454 th of a cubic foot, the weight of the specimen in grams is equivalent to its unit weight

in pounds per cubic foot. During the process of compaction, precautions were taken to try to keep the remaining material at its initial water content to insure that all specimens of a certain series would have relatively consistent moisture contents.

After completion of compaction and extrusion the specimens were prepared for preservation to maintain their original moisture contents during their required curing times. The procedure consisted of carefully wrapping them first in saran wrap and then in aluminum foil. The combination of saran wrap and aluminum foil was needed because of the tendency of some chemicals to react with the aluminum foil causing damage to the specimens during curing. Now the specimens were dipped in a molten micro-crystalline wax to form a wax coating of about 1 / 16 th inch thick. The indentification labels were attached to the specimens which were than placed in open trays and stored in the humid room for their respective curing periods.

The humid room was maintained relatively constant at about 70% humidity by a humidifer. The temperature of the humid room was also fairly constant due to the heating and air conditioning systems of the laboratory area.

Testing was performed with a Karol-Warner Model 550 Unconfined Compression Machine equipped with a Model KW-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 rate of loading to be varied.

The specimens were removed from their preservation wrappings and centered in the machine. At the instant the loading head comes in contact with the specimen, the loading head was stopped and the chart recorder was set to a zero strain reading. A strain rate of 0.3 inches per minute was then used until the load plot on the chart recorder indicated that the maximum load had been exceeded. The load was released, the type of failure recorded, and the majority of the specimen recovered for use in the moisture content determination. The maximum load and the strain for that load were logged in the data files for determination of maximum stress and strain.

Precautions to keep the specimens moist while being tested were not considered necessary since the temperature of the cured specimens and the atmospheric temperature were approximately the same. Also the maximum time the specimens were exposed to the atmosphere for testing and weighing was three minutes, which was not considered significant in producing loss of moisture.

# PVC Swell Test

The Soil PVC ( potential volume change ) Meters as designed by T. W. Lambe of Massachusetts Institute of Technology (Lambe 18), were used to determine the swell pressures due to different soil mixtures at various initial water contents. These swell pressures are used as a measure of the swelling characteristics of the different soil mixtures.

After completion of the proportioning and mixing of the materials for the PVC test, compaction was started. The compaction was performed by compacting the soil into a ring to make a specimen 2.750 inches in diameter and 0.625 inches in thickness. The material was placed in three equal layers with five blows per layer being applied with a hammer, similiar to the standard proctor hammer, that delivers a 5.5 pound force through a one foot free fall.

The specimen top was trimmed to produce a level surface and a specimen thickness of 0.675 inches. The ring and specimen was weighed to determine its wet density, and part of the trimmings were recovered for determination of the initial moisture content.

The specimen was now placed in the PVC Meter device between two dry, porous stones and assembled so that an initial load of 200 psf. could be applied to the specimen with the proving ring. The specimen was then completely submerged with distilled water and the time and dial readings were recorded for the necessary times.

The swell pressures exerted in psf. at the end of two hours submergence was considered as the swell index of the soil specimen for its particular initial moisture content. The device was now disassembled and the soil specimen was recovered for determination of its final moisture content and dry density.

#### CHAPTER V

### TESTS AND RESULTS

### Pilot Studies

In this initial study, seventeen trace chemicals were selected and mixed with gypsum-clay mixtures of 10% gypsum-90% clay and 30% gypsum- 70% clay. The clay used was mechanically sieved through a No. 10 sieve and the gypsum through a No. 100 sieve. The chemicals used were selected after a study of published reports about the effects of trace chemicals on clay-lime mixes. No chemicals were selected that were dangerous to use or were considered to be expensive.

In this study, one percent of each of the trace chemical additives was introduced into each of the two sets of gypsumclay mixtures. Specimens were also prepared of gypsum-clay mixtures without a trace chemical for purposes of control. For each series five specimens as nearly identical as possible were prepared at an optimum moisture content of 17% for the 10%-90% gypsum-clay series and 15% for the 30%-70% gypsumclay series. These specimen groups were cured for curing times of 1 day, 7 days, and 28 days before they were tested in unconfined compression. The trace chemicals used and the results of the unconfined compression tests for the different cures are tabulated and presented in figure 4. From a study

of figure 4 based upon the increased early strength of the mixtures for one day and seven days, it was quite obvious that sodium hydroxide had the greatest effect upon the unconfined compression strength but that sodium bicarbonate and sodium sulfite also had noticeable strength increases. Further study shows that all sodium compounds produce some increase in strength which may be because of the smaller atomic diameters of the sodium ions that allow a greater interparticle attractive force to develop.

# Intermediate Studies

The intermediate studies were concerned with a more detailed investigation of gypsum-clay mixtures using the three trace chemicals selected from the pilot studies. Here again 10%-90% gypsum-clay and 30%-70% gypsum-clay specimen mixtures were used.

In figure 5 is shown the results of using 0.5%, 1.0%, and 2.0% contents of sodium bicarbonate, sodium hydroxide, and sodium sulfite with the two gypsum-clay mixtures for 1 day, 7 days, and 28 days of curing time. Here again, it was quite evident that sodium hydroxide mixtures are associated with large increases in unconfined compression strength.

To study the effects of gypsum-trace chemical additives, a series of specimens were compacted using clay without gypsum and 0.5%, 1.0%, and 2.0% of the three trace chemicals for cures of 1 day, 7 days, and 28 days. These results are shown

in figure 6. Also in figure 6 are shown the control values of the gypsum-clay mixtures used here.

Comparison and study of figures 5 and 6 show: a definite superiority of sodium hydroxide over sodium bicarbonate and sodium sulfite. The use of sodium hydroxide with the gypsumclay specimens produces strengths much greater than those obtained in the clay specimens in which sodium hydroxide was used without gypsum. This could possibly indicate that a chemical reaction in the form of an ion exchange, either anionic or cationic, takes place between the gypsum and the sodium hydroxide. Also crystal growth was present upon many of the sodium hydroxide-gypsum specimens which also indicates an ion exchange of some type. The use of clay and sodium hydroxide also produced greater strengths then was obtained from the clay with gypsum tests.

Strengths obtained by the use of sodium bicarbonate and sodium sulfite were not noticeably affected either by the percentages of chemicals used for by the percentage of gypsum present in the clay soil specimens.

Further study also indicates a trend that the strengths obtained from the use of sodium hydroxide in clay with gypsum specimens increases as the percent of sodium hydroxide was increased up to 2.0%, as was done in this series of tests. This strength increase also parallels the results obtained from those specimens containing no gypsum.

### Sodium Hydroxide Studies

From the previous studies it was concluded that sodium hydroxide was the most effective of the trace chemicals investigated. At this point a thorough investigation of the use of sodium hydroxide as an effective gypsum-Permian Red clay stabilizer was launched.

For this series of tests, specimens of gypsum contents of 0.0%, 5%, 10%, 20%, and 30% were prepared with sodium hydroxide contents of 1%, 2%, 3%, and 5% at optimum moisture contents. Within the 2% sodium hydroxide groups, specimens were prepared at 2% above and below optimum. Specimens were prepared also for the 20% gypsum-80% clay-5% sodium hydroxide group at moisture contents 2% above and below optimum. Five identical specimens were prepared for each of the above combinations for curing periods of 1 day, 7 days, 28 days and 90 days.

The results of these tests in terms of unconfined compression strength are presented in figures 7 through 21. These figures show the effects of varying the quantities of sodium hydroxide, gypsum, and initial moisture.

Study of figure 7, which is clay without gypsum being present, indicates proportional strength increases as the amount of sodium hydroxide and the length of cure are increased. Comparison of figure 7 with figures 8 through 11 also shows this proportional strength increase for almost every point. Also figures 8 through 11 show that as the amount of gypsum is increased, there is a relatively constant increase in strength. This increase is shown in figure 11, for 30% gypsum,to be as much as 1.5 to 3 times the strengths obtained in figure 7 where gypsum was omitted. For low gypsum contents, the effects of increasing sodium hydroxide contents on the strength are erratic for 1 and 7 day cures, but are very consistent for 28 and 90 day cures. Therefore, some time must be required for the complete strength reaction of sodium hydroxide and gypsum to transpire.

For gypsum contents up to 30% and sodium hydroxide to contents of 5%, no optimum condition is seen to be evident.

In figures 12 through 15, the optimum percentages of gypsum for sodium hydroxide contents of 1%, 2%, 3%, and 5% are 13%, 13%, 20%, and over 30%, respectively. The amount of strength increase in all cases seems to be due more to the increased curing time then due to a particular content of either gypsum or sodium hydroxide. Up to 3% sodium hydroxide, a gypsum content of about 15% would seem to be about optimum and the amount of strength developed would be controlled by the length of curing allowed.

In figures 16 through 20 are shown the values and curves obtained from the 2% sodium hydroxide series of specimens which were prepared at approximately optimum, at 2% above, and 2% below optimum for the gypsum-clay mixture being prepared. This was done to study the effect and relation of the initial water content upon the strength of the different gypsum-clay mixtures.

Due to the variance of the plotted points, the curves

could not be drawn exactly, but were drawn to show what was believed to be the general trend of the points. The trend here seemed to be that moisture contents between 15% and 19% usually produced the highest strengths. These varied from about 18% for clay without gypsum and the lower gypsum contents (up to 20%), to about 15% for the 30% gypsum content.

Figure 21 shows the effect of initial moisture content for the Permian clay-20% gypsum and 5% sodium hydroxide. Here much higher strengths are produced due to the 5% sodium hydroxide, but moisture contents lower than 18% (shown in figure 19 for 2% sodium hydroxide), are required for the higher strengths.

These general curves, in figures 16 through 21, also show the proportional increase in strength due to the length of cure and the amount of gypsum that was also indicated in figures 7 through 15.

The effect of sodium hydroxide content upon the dry densities obtained from a given compactive effort upon mixtures of Permian clay and gypsum are shown in figure 22. In general as the percentage of gypsum was increased, the dry density became lower without regard to the sodium hydroxide content. Now if all cases are considered as one, a sodium hydroxide content of 2% to 3% would give about the greatest dry densities that could be obtained from these mixtures.

### Calcium Hydroxide Studies

Calcium hydroxide (lime) was used to study the effects and relationship developed by the use of small percentages of calcium hydroxide as compared to sodium hydroxide. Much work has been done on the use of calcium hydroxide as a soil stabilizer and is preferred in field work because of its ease of handling over that of sodium hydroxide. For these tests, a series of specimens were prepared for 20%-80% gypsum-clay mixtures for 1, 7, 28, and 90 days curing times. Five replicages were made for each combination, using calcium hydroxide percentages of 1%, 2%, and 5%.

Comparison of figure 10 with figure 23 shows that the curves produced by the two chemicals are quite similiar. The calcium hydroxide shows strengths a little greater than the sodium hydroxide strengths as the percent of chemcials and the length of curing time are increased.

In figures 24 through 26 are shown the effects of various admixtures of gypsum, sodium hydroxide, and calcium hydroxide on the unconfined strength of Permian clay prepared and cured at optimum conditions for 7, 28, and 90 days. Here besides only mixtures of gypsum and clay, were also mixtures of gypsum and clay with either 2% sodium hydroxide or 2% calcium hydroxide contents. These curves show that gypsumclay-2% sodium hydroxide produces substantial strength increases over the use of gypsum-clay only (up to 30% gypsum); but that those mixtures in which 2% calcium hydroxide was used showed a slight strength increase over those mixtures in which 2% sodium hydroxide was used. In connection with the use of sodium hydroxide, the indications are that an optimum amount of gypsum occurs around 20 percent.

## Loess-Gypsum Studies

To investigate the effects of gypsum and sodium hydroxide upon a soil less plastic than the Permian clay, a loess material was obtained for study. Gypsum contents of 0, 10, 20, and 30 percent was used with loess contents of 100, 90, 80, and 70 percent, respectively without sodium hydroxide. Some specimens of 10%-90% and 30%-70% gypsum-loess combinations were prepared with 1 percent sodium hydroxide. Five identical specimens were prepared for 1, 7, and 28 days of curing for each of the combinations. The strength values obtained from these different combinations are shown in figure 27.

The use of increased amounts of gypsum seems to produce some proportional increases in the strengths for the 1 and 7 day cures; but the longer curing time of 28 days does not produce a marked strength increase.

The use of 1 percent sodium hydroxide with the 10%-90% and 30%-70% gypsum-loess mixtures produces strengths over twice those obtained from loess mixtures without either gypsum or sodium hydroxide. Greater strengths also resulted from the 30%-70%-1% gypsum-loess-sodium hydroxide combinations then from the 10%-90%-1% gypsum-loess-sodium hydroxide combinations. This seems to indicate a need of the sodium hydroxide for a larger amount of gypsum for a more complete chemical reaction.

From a comparison of figures 4, 12, and 27, the indications are that the use of loess (for curing periods up to 28 days) does not produce a noticeable strength increase or decrease over the strengths obtained from the use of Permian Red clay.

#### Fine Gypsum Studies

To study the effect of using gypsum finer then that passing the No. 100 sieve, a quantity of gypsum passing the No. 270 sieve was obtained. Specimens were prepared for fine gypsum contents of 0, 5, 10, 15, 20, 30, and 50 percent with Permian clay at optimum moisture contents and allowed to cure for 1, 7, 28, and 90 days.

The results, as shown in figure 28, indicate that the use of the fine gypsum has almost no effect upon the strength as compared to the strength of clay without gypsum. The erratic variation of the plotted points probably arises from the variation in moisture contents of the tested specimens, but was not believed to have affected the strengths obtained to any major degree. Also comparison with figures 25 and 26 shows very little difference in the strengths obtained using fine gypsum over those obtained using the normal gypsum. Therefore, no further studies were made in the use of the fine gypsum.

### Swelling Pressure Studies

As was discussed earlier, the PVC meter was used to measure the swelling pressures developed from confined specimens. Specimen combinations of 10%-90% gypsum-Permian clay, 30%-70% gypsum-Permian clay, 10%-90% sand-Permian clay, and 30%-70% sand-Permian clay were prepared and tested. A series of specimens were also tested using 1%, 2%, 3%, and 5% sodium hydroxide with the 10% and 30% gypsum contents. Also prepared were specimens using 2% and 5% calcium hydroxide instead of sodium hydroxide. These specimen series were all prepared at initial moisture contents of 6%, 11%, 15%, and 19% during compaction. For control, specimens were used of Permian clay without additives and Permian clay with the above mentioned percentages of sodium hydroxide and calcium hydroxide without gypsum.

As was dicussed in Chapter IV, the swell pressure measured at the end of two hours of confined submergence was considered to be the swell index. These values obtained for the above test combinations are shown in figures 29 through 36.

Study of figure 32 shows that as the initial moisture content of the Permian clay decreases, the swelling pressure increases. For a decrease to 10% initial moisture content, from an optimum of 18 percent for Permian Red clay, there is developed a swell pressure that is three times greater then at optimum. Figure 29 shows the swell pressures developed when 10% and 30% gypsum are combined with the Permian clay. This shows a decisive decrease in swelling when 10% gypsum is used and a further reduction when 30% gypsum is used. Comparison of figure 29 with figure 30 shows that the use of sand in the place of gypsum also decreases the swell pressure to almost the same values. This indicates that the reaction between the gypsum and Permian clay must be of a physical nature because of the almost identical results obtained from the use of sand which is a non-reactive substance. The use of gypsum and sand (up to 30%) both cause decrease in swell pressures from those caused by Permian clay alone for short periods of time.

Due to the relatively high percentages of montmorillonite and illite in the Permian Red clay the tendency of the clay to expand (because of the characteristic of expanding lattices) while in the presence of available water is considerable. This expansion of Permian Red clay (shown in figure 32) takes place at all initial moisture contents and is much greater at the low initial than at high initial moisture contents. Further expansion is caused by the double layer water deficiency which causes water to flow into the soil. This water flow continues until the repulsive pressures between the particles become equal to the applied effective stress.

To determine the swelling potentials after long periods of curing upon the reaction of gypsum-clay, a series of tests was performed on some gypsum-clay mixtures that had been cured for 18 months at initial moisture contents near the liquid limit. Figure 31 shows that for these gypsum contents of 18.4% and 46.5%, there was hardly a noticeable difference in the swelling pressures. Therefore it may be concluded that a chemical reaction between gypsum and Permian Red clay does not seem to exist.

In figure 32, is shown the results of adding 2% and 5% sodium hydroxide to Permian clay without gypsum. The sodium hydroxide causes a small reduction in swelling potential but probably not enough to be of much use in practice.

Figures 33 and 34 show the effects of adding different percentages of sodium hydroxide to clay mixtures with gypsum contents of 10 and 30 percent. These figures show that there is an increase in swell pressures as the percentage of sodium hydroxide is increased, and also as the percentage of gypsum is increased. This increase with gypsum content is the reverse of figure 29, where increased gypsum content led to a decrease in the swell pressure. Up to 3% sodium hydroxide, the swell pressures developed are below those produced by clay but above those when gypsum was added to the clay. The use of 5% sodium hydroxide produces pressures considerably greater than any other combination of materials. This would probably be very detrimental if used in practice. The reaction of sodium hydroxide with the gypsum must therefore be a chemical one that causes a volume change. Therefore increased amounts of sodium hydroxide and gypsum would tend to cause increasing volume changes which would affect the swelling pressures developed.

Use of 2% and 5% calcium hydroxide in place of sodium hydroxide is shown in figures 35 and 36. The use of calcium hydroxide for all combinations produced swell pressures that were less then the swell presures developed when clay was present without additives (figure 32). For both percentages of calcium hydroxide a very noticeable reduction in swell pressures occured for initial moisture contents above 10 percent as compared to the swell of clay alone. Further reduction was caused by the addition of gypsum, and more reduction as the gypsum content was increased. A definite reduction took place for initial moisture contents above 10 percent as the percentage of calcium hydroxide was increased. For moisture contents below 10 percent, the reduction due to increasing the amounts of calcium hydroxide was slight if any. Below 10 percent the large reduction in swell pressure seemed to be due to the presence of gypsum only.

The effect of increasing the percentages of calcium hydroxide and gypsum (which results in swell pressure reductions as contrasted to the opposite effects caused from the use of sodium hydroxide and gypsum) indicates that a chemical reaction does not occur (during this short test time) between the calcium hydroxide and gypsum. However, it is known that calcium hydroxide will react with the clay, even though considerable time may be required for completion of the reaction.

# CHAPTER VI

#### CONCLUSIONS AND RECOMMENDATIONS FOR FUTURE RESEARCH

# Conclusions

This investigation was conducted for the purpose of determining if sodium hydroxide used as a trace chemical additive with Permian Red clay-gypsum soils would be an effective soil stabilizer. The investigation was based upon the use of the unconfined compression strength test and the potential volume change (PVC) test. From the investigation dicussed in the preceeding pages the following specific conclusions can be made:

- (1) That sodium hydroxide (up to a content of 5 percent) increased the unconfined strength of the Permian Red clay (with up to 30 percent gypsum) increasing with increased curing periods.
- (2) An optimum gypsum content of approximately 15 percent is found to exist for sodium hydroxide contents up to 3 percent, with the strengths developed dependent upon the length of cure. No optimum gypsum content was reached for 5 percent sodium hydroxide.

(3) The best strengths were developed for all gypsumclay-sodium hydroxide mixes for initial moisture

contents of 15 to 19 percent.

- (4) A sodium hydroxide content between 2 and 3 percent produces the highest dry densities for these mixes.
- (5) The use of calcium hydroxide produces strengths that compare to those obtained using sodium hydroxide.
- (6) The use of sodium hydroxide with loess-gypsum mixes was not very effective in producing strengths more than twice those obtained from loess alone, and also was not effected by the length of cure.
- (7) The use of gypsum finer than No. 270 size was not much better than using gypsum passing the No. 100 sieve.
- (8) The use of increasing amounts of gypsum or sand (up to 30 percent) is quite effective in reducing the swelling of the Permian Red clay.
- (9) The use of sodium hydroxide upon the Permian Red clay was not very effective in reducing the swelling pressures developed by specimens compacted immediately after mixing.
- (10) The use of sodium hydroxide with gypsum allows greater swell then using gypsum alone but is still quite effective in reducing swelling as compared to not using gypsum or sodium hydroxide at all. From the specific conclusions listed, a general conclu-

sion about the use of sodium hydroxide as an effective stabilizer is:

That the use of 2 to 3 percent of sodium hydroxide

with a gypsum-clay mixture of 15% gypsum- 85% Permian Red clay compacted at an initial moisture content of 15 to 19 percent is a very effective stabilizer. This combination of materials produces suitable densities, very good strength increases and good swell reductions.

Recommendations for Future Research

The following listed items are recommendations that may be considered for further investigations of the effectiveness of sodium hydroxide on gypsum-clay combinations:

- (1) Controlled field testing should be performed and investigated to determine the correlation with the laboratory investigations.
- (2) Economic studies should be conducted upon the practicality of using gypsum and sodium hydroxide as clay soil stabilizers.
- (3) Further refinements of the PVC test should be investigated to be able to reproduce more consistent test results.
- (4) Corrosion effects of using the different chemicals upon the laboratory and field equipment.
- (5) Storage and handling problems associated with the use of chemicals in soil stabilization should be investigated.



FIGURE I

GRAIN SIZE DISTRIBUTION CURVE FOR PERMIAN RED CLAY



FIGURE 2

GRAIN SIZE DISTRIBUTION CURVE FOR GYPSUM



FIGURE 3

GRAIN SIZE DISTRIBUTION CURVE FOR LOESS



## U/C Strength (<sup>tons</sup>/sq. ft.)

Figure 4

Effect of Various Trace Additives on the Strength of PRC - Gypsum Mixes

5 W





Effect of Selected Trace Additives on the Strength PRC - Gypsum Mixes



Figure 6

Effect of Selected Trace Additives on the Strength of Permian Red Clay



Compressive Strength of Permian Clay



Effect of Sodium Hydroxide on Strength PRC - Gypsum (5%) - NaOH Optimum Series











Figure 11

Effect of Sodium Hydroxide on Strength PRC - Gypsum (30%) - NaOH Optimum Series





Effect of Gypsum on the Strength of Mixtures of Permian Clay with 1% Sodium Hydroxide





Effect of Gypsum on the Strength of Mixtures of Permian Clay and 2% Sodium Hydroxide





Effect of Gypsum on the Strength of Mixtures of Permian Clay and 3% Sodium Hydroxide

A Maria





Effect of Gypsum on the Strength of Mixtures of Permian Clay and 5% Sodium Hydroxide




Effect of Moisture Content on the Strength of Permian Clay with 2% Sodium Hydroxide



Gypsum (5%) - NaOH (2%)





Gypsum (20%) - NaOH (2%)







Gypsum (20%) - NaOH (5%)











Optimum Series - Cured for 90 Days



Effect of Various Admixtures on the Unconfined Compressive Strength of Permian Clay

Optimum Series - Cured for 28 Days





Effect of Various Admixtures on the Unconfined Compressive Strength of Permian Clay

Optimum Series - Cured for 7 Days









Effect of Fine Gypsum on the Unconfined Strength of Permian Clay



Swelling Pressure of Mixtures of Permian Clay and Gypsum







Swelling Pressure of Mixtures of Permian Clay and Gypsum After 18 Month Curing



Pressure of Permian Clay













### BIBLIOGRAPHY

- (1) Woods, K. B. and Yoder, E. J., "Stabilization with Soil, Lime, or Calcium Chloride as an Admixture," <u>Proceedings of the Conference on Soil Stabilization</u>, Massachusetts Institute of Technology, p. 3, June 18-20, 1952.
- (2) "Gypsum Treated Soils Do Not Expand," <u>Rock Products</u>, Vol. 63, No. 7, p. 10, 1960.
- (3) Davidson, D. T. and Mateos, M., "Evaluation of Gypsum as a Soil Stabilizing Agent," <u>Soil Stabilization</u> <u>with Chemicals</u>, Iowa Engineering Experiment Station, Iowa State University, Bulletin No. 193, pp. 338-340, 1960.
- (4) Matalucci, R. V., "Laboratory Experiments in the Stabilization of Clay with Gypsum," <u>Master's</u> <u>Thesis</u>, Oklahoma State University, 1961.
- (5) Bowman, J. W., "The Effects of Gypsum on the Strength of Clay," <u>Master's Thesis</u>, Oklahoma State University, 1963.
- (6) Mack, J. R., "Gypsum, Anhydrite and Plaster of Paris as Soil Stabilizers," <u>Master's Thesis</u>, Oklahoma State University, 1963.
- (7) Winterkorn, J. F., "The Science of Soil Stabilization," Soil and Soil-Aggregate Stabilization, Highway Research Board, Bulletin No. 108, p. 1, 1955.
- (8) Murray, G. E., "Soil Stabilization by Chemical Means," <u>Proceedings of the Conference of Soil Stabilization</u>, <u>Massachusetts Institute of Technology</u>, pp. 107-108, June 18-20, 1952.
- Wooltorton, F. L. D., "Engineering Pedology and Soil Stabilization," <u>Soil and Soil-Aggregate Stabilization</u>, Highway Research Board, Bulletin No. 108, p. 50, 1955.
- (10) Hauser, E. A., "Soil Stabilization and Colloid Science," Soil and Soil-Aggregate Stabilization, Highway Research Board, Bulletin No. 108, pp. 58-64, 1955.

- (11) Means, R. E. and Parcher, J. V., <u>Physical Properties of</u> <u>Soils</u>, Charles E. Merrill Books, Inc., pp. 20-29, Columbus, Ohio, 1963.
- (12) Grim, R. E., Clay Mineralogy, McGraw-Hill Book Co. Inc., York, Pa., pp. 126-134, 1953.
- (13) Ibid, pp. 156-158.
- (14) <u>Ibid</u>, pp. 161-163.
- (15) Lambe, T. W., Soil PVC Meter, Federal Housing Administration, Bulletin No. 701, Washington, D. C., pp. 5-18, December, 1960.
- (16) Op cit, Murray, G. E., pp. 112-114.
- (17) "Gypsum," <u>Industrial Minerals and Rocks</u>, 3rd. ed., The American Institute of Mining, Metallurgical, and Petroleum Engineers, Maple Press Company, York, Pa., pp. 471-473, 1960.
- (18) Op cit, Lambe, T. W., pp. 21-29.
  - Parcher, J. V., "Soil Stabilization Project Final Report," School of Civil Engineering Research Publication, Oklahoma State University, Stillwater, Oklahoma, No. R-22, 1964.
  - Yoder, E. J., <u>Principles of Pavement Design</u>, John Wiley and Sons, Inc., New York, 1959.
  - Soil Stabilization Studies, Highway Research Board, Bulletin 183, 1957.
  - Stabilization of Soils, Highway Research Board, Bulletin 98, 1954.
  - Soil Stabilization, Highway Research Board, Bulletin 69, 1953.
  - Lime and Lime-Flyash Soil Stabilization, Highway Research Board, Bulletin 193, 1958.
  - Chemical and Mechanical Stabilization, Highway Research Board, Bulletin 129, 1956.
  - <u>Influence of Stabilizers on Properties of Soils and</u> <u>Soil-Aggregate Mixtures</u>, Highway Research Board, Bulletin 282, 1960.
  - Preconditioning and Stabilizing Soils by Lime Admixtures, Highway Research Board, Bulletin 262, 1960.

# VITA

# Maurice Leroy Mitchell

### Candidate for the Degree of

# Master of Science

### Thesis: SODIUM HYDROXIDE AS A PERMIAN CLAY-GYPSUM STABILIZER

Major Field: Civil Engineering

Biographical:

Personal Data: Born at Columbus, Kansas, September 4, 1940, the son of Claude Allen and Alta Louise Mitchell.

- Education: Attended grade schools in Southern Kansas and throughout Oklahoma; graduated from Heavener Senior High School at Heavener, Oklahoma in May 1958; received the Bachelor of Science degree from the Oklahoma State University, with a major in Civil Engineering, in May 1963; completed requirements for the Master of Science degree at Oklahoma State University in Civil Engineering, in May, 1964.
- Professional Experience: Summer employment with H. D. Youngman Contractor, Inc. for summers of 1958 through 1963; Student Member of American Society of Civil Engineers; Junior Member of National Society of Professional Engineers; Junior Member of Oklahoma Society of Professional Engineers; Member of Chi Epsilon Fraternity; Engineer-in-Training(State of Oklahoma).