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# THE UNIVERSITY OF OKLAHOMA GRADUATE COLLEGE

# STRENGTH AND CONSOLIDATION PROPERTIES OF RAW AND STABILIZED OKLAHOMA SHALES

A DISSERTATION

SUBMITTED TO THE GRADUATE FACULTY

in partial fulfillment of the requirements for the

degree of

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BY THOMAS JOSEPH ANESSI Norman, Oklahoma

STRENGTH AND CONSOLIDATION PROPERTIES OF RAW AND STABILIZED OKLAHOMA SHALES

APPROVED BY

DISSERTATION COMMITTEE

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# STRENGTH AND CONSOLIDATION PROPERTIES OF RAW AND STABILIZED OKLAHOMA SHALES

# CHAPTER I

# INTRODUCTION

Throughout Oklahoma, there is a variety of soil materials known as shale. Each farmer, geologist or highway engineer seems to have his own definition of shale based on his own experience with it. Its range of attributes seems to run the gamut from the highly cemented rock-like shales which are used as paving aggregates to non-compacted clays and mudstones whose established orientation through sedimentation and long term consolidation, at least technically, qualifies them as shales, despite the fact that they would serve poorly as construction materials.

The foregoing infers that shales might have a variety of engineering properties, from the acceptable to the unacceptable. Both of these extremes are quite easily determined through standard tests, long established in engineering practice. As long as these tests, which treat rock-like shales as rock and soil-like shales as heavy clays, seem sufficient to predict the response of these

materials throughout their design life, little additional investigation might be required.

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However, for many years, more simplified procedures have not been completely successful in dealing with all the shales. Changes within the materials brought about by changes in their environment have resulted in failures to thousands of structures throughout the United States. By its very nature the performance of shale is affected by its moisture content, its cementing constituents, the clay minerals present, and the loads imposed on it through its life.

Particularly significant to members of the Oklahoma Department of Highways was that the variation of these factors with time seemed to result in considerable, and often critical changes to the engineering properties observed. This situation was termed, for want of a better description "weathering."

Simply put, this "in-between" type of shale, which, when first observed, had desirable physical properties, often weathered quickly to the point where it was no longer acceptable, that is, capable of fulfilling its design function.

Correspondence with 47 state highway departments indicated that considerable experience in this area was gained during the recent decade (1). Rising construction and material transportation costs have necessitated making greater

use of available shale materials. Although it became increasingly apparent that great care be taken in predicting those properties of shales which would make them perform undesirably in the field, many failures both to backslopes and to subgrades resulted from unexpected high weathering alterations.

As a result of this experience, the Oklahoma Department of Highways obtained the services of the Oklahoma University Research Institute to determine a method of predicting the weathering response of similar shales throughout the state (2). The numerous samples obtained through this study provided a source of shales whose physiochemical and engineering characteristics placed them in the highly "weatherable" category; shales which originally could be expected to be used in construction with little or no modification but which when weathered, failed to measure up to design specifications.

Since the use of lime stabilization to modify clays is an established practice in Oklahoma, its usefulness in curbing or simply delaying the weathering of these shales, seemed a natural and appropriate step. Lime had been used for many years to reduce soil plasticity and to otherwise improve the soil workability during construction. Further gains had been realized through reduction of shrinkage and sizable strength increases. This last benefit, strength increase, has been given more and more attention in recent

years by researchers. Applications of lime to clay soil subbases and bases of 4 to 12 inches in depth have been quite common throughout the United States. Recent development of 24 inch treatments in Oklahoma have also met with considerable success.

One might conclude that most of the soils treated as described above, because of their relatively high plasticity, contain large amounts of clay. Except for the lack of particle orientation or lamination, these soils are quite similar in particle size distribution to a material such as shale as defined geologically. It is noteworthy that many of these soils, though not well laminated, have been referred to as shales in the past, depending on the experience and judgment of the investigator.

However, past studies have not included any appreciable investigations into shale-lime stabilization. Rather, they have been made on soils whose clay fractions are made up of distinct particles. The case where the particles, or at least a sizable number of them, are still "clustered" and in a possibly well oriented condition, and in which some weathering response through physical or chemical breakdown might yet be expected, has not received much attention.

The purpose of this dissertation is to investigate how six of these typical Oklahoma shales react to lime stabilization and how these reactions might be explained in the light of contemporaty engineering theory.

# CHAPTER II

# BACKGROUND

# Shales in General

All definitions of shales contain the same basic terms, "laminated," "sedimented," "clay particles" (3). Shales are likely to include anything indurated, fissleable, or laminated, with the degree or limit of any of these terms quite open to question. The material tends to break along planes approximately parallel to the bedding planes although some secondary breakage can occur as a result of vertical pressures exerted by overburden.

From this point, the exact application of the term "shale" to any particular soil mass becomes even more clouded. Many differences of opinion have arisen as to the constitution of the material. Some authors such as Terzaghi (4), classify shales as rock-like, that is, able to cause a ringing sound when hit by a hammer. Underwood (3) considers shale to include a highly indurated and generally fissile equivalents of claystone and/or siltstone. Many engineers are even inclined to discount fissility as a

requirement and so to apply the title to heavily compacted laminated clays or silt clays.

The use of the term "fissility," itself introduces difficulty since one must determine the degree of fissility involved. Ingram (5) notes three types of fissility; massive, flaggy, and flaky, which inclines acceptance of mudrocks of questionable degrees of lamination, since massiveness itself implies random particle arrangement.

Underwood's broad attempt to classify shales includes a classification of sedimentary rocks (see Figure 1) which identifies shales as compaction or "soil-like" and cemented or "rock-like." A distinguishing test between the two is that the former tends to slake rapidly in water while the latter does not (3). This intimates that the rock-like shale is well cemented.

It would seem appropriate to note that the shales in this study, though generally soil-like in nature, do not conveniently fall into either category described, but fall into a category which might be called "partially cemented" shales. In effect, these shales, though they meet the chemical composition normally proposed by most writers (6) for shales, vary in their susceptibility to weathering by their degree of cementation as well as by their mineralogy and degree of consolidation.



Figure 1 . Classification of Sedimentary rocks, after Underwood.

Engineering Properties of Shales Atterberg Limits (plasticity)

The standard methods employed to test soil plasticity as measured by the Atterberg Limits (plastic limit, liquid limit, and plasticity index), have special significance in the case of shales since it is dependent on the state of the material at some point between the indurated condition in which it is initially found and the fully brokendown or weathered condition. Standard preparation procedures as defined in AASHO (7) and other publications often result in doubtful values, particularly when these values are compared with those obtained for the same shales after they have been subjected to moderate weathering (2).

Recent tests at the University of Oklahoma, School of Civil Engineering and Environmental Science, using ultrasonics as a means of obtaining optimal breakdown of shales, indicate that increases of 0 to 15 in liquid limit and plasticity index values are obtained when the shales are subjected alternately to standard and ultrasonic preparation methods (8). Although the full implications of this research have not been realized at the time of this writing, the dependence of the Atterberg Limits on the method of specimen preparation, or more specifically on the status of weathering, appears conclusive.

### Strength

The strength characteristics of laminated, compacted and often, partially cemented clay shales, vary greatly depending on the amount of compaction, type and amount of cementing material, particle orientation and the existing moisture conditions. Under unexcavated conditions, even soil-like shales have extremely high compressive strengths compared to weathered or remolded strengths. This "weakening" appears to be due to energy releases caused by loss of consolidation pressures, modification of particle orientations, creation of random particle orientations, or loss of cementing bonds which result from weathering or from field sampling or laboratory preparation procedures. Likewise, reductions in shear strengths seem likely to occur whenever natural shales are similarly disturbed (9).

# Moisture-Density

The natural moisture contents and densities of shales are dependent on the weathered conditions in which they exist. When undisturbed and well compacted, their moisture contents might be quite low and densities quite high. Underwood (3) presents natural moisture content values in the range of 5 to 35 percent and densities of 80 to 160 pcf. The higher moisture contents and lower densities of soil-like shales generally conform to those experienced in highly plastic, poorly consolidated clays. Underwood (3)

further suggests that troublesome foundation shales might occur whenever their natural moisture content exceeds 20 percent.

### Void ratio and permeability

The values of void ratio for soil-like shales usually conform to those obtained for compacted clays of the same particle size distribution. These shales might be expected to experience a considerable increase in void ratio upon the removal of loading.

Rock-like shales differ in that any cementing action interfers with the consolidation and rebound response of soil particles. The effect of cementing materials on void ratios might vary considerably. In certain cases they might tend to fill voids within the soil mass and thereby decrease the volume of voids. On the other hand, cementing materials in poorly consolidated clays might result in a lattice-like structure which actually support larger void volumes. In either case, increases or decreases in void ratios as a result of load variations are usually much lower for rock-like shales than for soil-like shales because of the structural contributions of the cementing materials.

The permeabilities of shales are usually quite low and difficult to measure because of the high amount of clay present and its consolidation status. Varved conditions tend to increase permeability values considerably and also

increase the amount of water available to less permeable layers (3).

# Swelling and shrinkage

The swelling and shrinkage of shales vary from practically none in the case of the rock-like variety to extremely high values for many soil-like materials. The amounts of water adsorbed by clay minerals is dependent on the type and amount of clay minerals present, the amount of preconsolidation, the availability of water and the type of cementing materials present in the soil mass.

The effect of mineralogy on the expandability of clays is well known by soil scientists. The publications of Grim (10) serve as a fairly typical evaluation of this property. Colloid size particle surfaces attract and hold water by electrostatic means, depending on the type of clay mineral involved. Montmorillonites have been selected as the most troublesome clay mineral for engineering use with illites and kaolinites usually picked as culprits of a lesser breed. Certain varieties of montmorillonites (i.e. lithium and sodium) have recorded extremely high expansions in the order of 400 percent and more, whereas, at the other extreme, the more crystalline kaolinites have exhibited little or no tendency to swell.

Likewise, considerable work has been done to relate the amount of swell in a soil to the amount of clay present (11).

Preconsolidation and cementing serve to discourage the effects of water on the clays by physically impairing the availability of water to the particle surface or the mineral interlayers.

Preconsolidation performs its function primarily by strengthening the electro-chemical bonds between particles through providing orientation and establishing interfacial contacts between particles. These result from extremely high pressures and from the subsequent forcing out of water from the soil mass.

Cementing materials, on the other hand, tend to coat the constituent particles, form bridges between them or fill voids which provide water accesses between them, thereby interferring with the normal expansive process experienced when the clay mineral comes in contact with water.

Weathered shales might be considered to be those from which significant amounts of cementing materials have been leached out and/or in which interparticle bonds and orientations have been reduced sufficiently to allow significantly increased clay mineral expansion when the shale is brought into contact with water.

### <u>Activity</u>

Skempton introduced (12), and Seed et al. (13) modified a measure of "activity" of clay soils. This has been referred to as the activity index and, as most often used, is computed according to the following formula by Skempton:

or according to the following formula by Seed et al.:

Several authors, including Underwood (3) have determined that clay soils containing montmorillonite or illite as their predominant clay mineral constituents have the highest activity index. Some care must be taken, however, in interpreting the results of activity indices since comparisons between shales could turn into an unproductive "numbers game." Results seem to vary drastically when computed by using either low P.I. values or low percentages of 2 micron clays.

# <u>Consolidation</u>

Consolidation information currently available in most publications is normally concerned with clay soils or soil-like shales rather than with rock-like shales, or soils of elastic rather than plastic response to loading, inasmuch as the latter are inclined towards more predictable and less troublesome behavior when subjected to loading conditions associated with highway and highway structure construction. (A response to recent inquiries of 47 state highway departments indicated that designs involving "trouble" shales were usually handled in a similar manner to those involving common clays (1).)

The consolidation phenomenon is considered to occur as soil particles are forced together during continued and increased loading of the overlying soil or structural layer. This action is accompanied by a gradual reduction in water content as mobilized pressures force excessive liquid out of the soil mass (14). This effectively reduces the void ratio (that is, the ratio of the volume of the voids to the volume of the soil solids) at any point in the soil mass. Consolidation is similar to compaction except that it involves much greater periods of time.

The energy involved in consolidation normally consists of both structural forces and electronegative forces between particles, the first resulting in elastic response and the second in repulsive response.

Within clays, consolidation is affected greatly by the orientation of particles within the soil mass and by the low permeabilities normally associated with these materials.

The opposite of consolidation has been referred to as swelling, expansion or rebound and involves reduction in loadings with decrease in resulting pressures, reversal of dewatering activities, increase in void ratios and decrease in dry densities.

The theory of consolidation is complicated somewhat by its phasing, often described as "primary consolidation" and "secondary consolidation." Practical differentiation between the two phases is difficult since the two occur

simultaneously. The former is usually credited with the larger initial reduction of the soil volume caused by forcing particles closer together and concurrent water explation. The second is associated usually with considerably smaller volume reductions caused by slippage between particles and resistance to shear deformation due to highly viscous absorbed water (14). Nearly all standardized tests currently in use involve determination of consolidation properties dominated by the primary stage, and most engineering design seems to assume that secondary consolidation values are insufficient to cause great concern except under very special design circumstances.

The application of consolidation theories to shales becomes involved because in shales the following should also be considered: the previous consolidation history of the clay, the effects of sample disturbance and the effects of cementing materials, and salts or other solubles on the shale structure.

# Preconsolidation

Preconsolidation involves maximum load intensity to which a soil has been subjected during its lifetime. This load might result from high surcharge pressures which, in Oklahoma might reflect soil and/or water heights of up to 7000 feet, even in the case of shales now located much closer to existing ground levels. This property is, in effect, a "residual" consolidation which finds particles in

much closer proximity than one would expect under more recent loading conditions. This occurs because rebound resulting from the removal of surcharge is a very slow process and complete rebound to any particular lesser loading condition is probably never attained by a plastic soil mass.

Casagrande has proposed (15) that the preconsolidation pressure value might be detected in typical void ratio vs pressure diagrams at approximately that point where the curve deflection rate is maximum and has suggested a method for its determination. Schmermann (16) has questioned the accuracy of laboratory determinations of preconsolidation pressures for specimens removed from the field and has defined an approximate method for obtaining a corrected preconsolidation pressure from disturbed specimens.

# Disturbance

A soil property known as the virgin curve is derived from the fact that all consolidation curves for a particular soil tend towards a uniform rate of decline as loading increases between the completely undisturbed and the completely remolded state (14). (See Figure 2) Many authors have therefore accepted the fact that virgin curves of a particular soil do not vary greatly despite differences in preconsolidation or degree of disturbance. Work by Schmermann confirms this, although he shows that laboratory results of consolidation tests on relatively undisturbed specimens of sensitive clays are not necessarily indicative



Figure 2. Effect of depth on consolidation characteristics of a soil.

of the soil response to field loading and that corrections are in order (16).

### Cementing materials

Cementation or recrystallization is one of the most critical factors in determining the response of shales to consolidation. The type of cementing material, its solubility, amount and reactibility with the mineral components in the soil all contribute to its ability to break down on loading or on weathering.

Cementing materials might be calcareous, siliceous, ferruginous, gypsiferous, phosphatic, etc, and cause modification to the shale by recrystallization of the clay minerals or by establishing other diagenetic bonds (17).

The fact that a certain amount of cementing material is present in all soils is in great part, responsible for the essentially empirical nature of the consolidation test itself and its influence has been given little concentrated attention in past engineering studies.

# Salts and solubles

Saline soils are usually found in areas which are formed from transported soils from other locations such as marine deposits. Shales are perhaps the most common example of such deposits (18).

The most common salt-forming components (ions) in the earth's crust are sodium, calcium, magnesium, chloride, and

sulfate. Carbonates and bicarbonates are involved in the weathering of soils. Bicarbonates form from solutions of carbon dioxide in water and constitute a particularly effective weathering medium. The relative amount of carbonates and bicarbonates is a function of pH. Carbonates usually require a pH of 9.5 or more to exist in quantity.

Salts and other solubles provide the ions which affect the clay soil character through any one of the following ways:

- 1. exchange with other ions within the basic clay structure.
- 2. exchange or adsorption in the interlayer.
- 3. exchange or adsorption on the particle surface.
- 4. presence in the soil water.
- 5. combination with other ions to form other compounds such as cementing agents.

All of the foregoing factors, in addition to affecting the response of shales to consolidation loads are themselves affected in varying degrees by weathering when exposed. Such occurrences as separation or disorientation of particles, reduction of electrostatic bonds, simple disturbances, and leaching of cements, ions, or salts, might result in drastic changes to response of a soil to consolidation pressures.

# Lime Stabilization of Soils

# General

The use of hydrated lime (hereafter referred to as "lime") as a soil stabilizer in United States roadway construction has significantly increased during the past three decades. So many authors and organizations have attempted to describe the historical progress of its use that an additional attempt would only serve to repeat already commonly available information (17).

The effects of lime as a stabilizer are also generally well known and may be grouped into the different property modifications which it normally is expected to bring about:

- 1. short term reduction of plasticity,
- 2. short term reduction of swelling,
- 3. short term reduction of permeability,
- 4. long term increase in strength.

Such modifications have made lime an ideal stabilizing agent for highway bases and subbases constructed in clayey soil areas. Although the fact that such modifications occur is obvious and that their values are measurable, several authors including Diamond and Kinter (19) reflect the chagrin of many engineers at the fact that very little is known about the details of the reaction processes involved in lime stabilization. They suggest that most of our information is only speculative in nature, despite a considerable number of investigations. This is not to say that much light has not already been shed on these details especially during the past decade, for despite some conflict between "schools of thought" on the exact method by which lime and soils react, a fairly clear understanding of its nature exists among engineers and soil scientists (19, 20, 21).

Diamond and Kinter (19) presented an "interprative review" of the soil-lime mechanism describing the physicalchemical reactions which take place when the two materials are mixed in the presence of water.

Certain mechanisms expected for long periods of time to account for the stabilizing effect of lime on soils, were critically discussed. Although their existence was not denied, several of these mechanisms were either relegated to minor roles in the process, or discounted completely, since they failed to adequately explain the stabilizing phenomenon. For example:

1. Cation exchange.--The replacement by limederived calcium cations of other exchangeable cations, either in part or when carried to complete saturation has been credited with causing flocculation of clays into silt sized particles which effectively reduced soil plasticity. Iowa State investigators (20, 21, 22) have proposed that naturally hydrophobic particles become hydrophillic when brought in contact with certain cations.

2. Clay flocculation.--As typically defined, clay flocculation intimates that a clay will flocculate on the addition of an electrolyte because of the modifying effect of the electrolyte on the extension of the electrical double layer from the surfaces of the clay particles. The electrolyte represses the double layer and thus reduces the electrostatic repulsive forces between clay particles resulting in a net attraction (23).

3. Carbonation.--The creation of cementitious bonds between particles from the reaction of lime with  $CO_2$  and water.

All of these theories attempt to explain primarily the first of two stages of soil-lime reaction, that short term or rapid process often called amelioration which results in the following typical effects on the original clay soil:

- 1. immediate increase in plastic limit.
- immediate modification of liquid limit, (usually decrease).
- 3. decrease in plasticity index.
- 4. increase in effective grain size of constituent particles.
- 5. reduction in shrinkability and expandability.
- 6. increase in moisture content and decrease in density for the same compactive effort, and
- 7. decrease in permeability of the compacted soil.
The carbonation of lime by  $CO_2$  from the surrounding air was at first thought to be partially responsible for both immediate and long term strength increase of the soil mass, but later experimentation indicated that only very weak cementation was obtained, far weaker than that required to explain the new strengths obtained (24).

# <u>Short term (amelioration)</u> <u>effects</u>

Short term effects of lime soil mixtures are those expected during the first hour of mixing. These effects are normally produced by the addition of small percentages of lime, usually no more than 3 percent by dry weight of the soil fraction. In the past, that amount of lime required to produce all of the amelioration effects noted above without providing for long term strengthening has been referred to, perhaps mistakenly, as the "lime fixation point" or "lime retention point" (22, 25, 26).

Several researchers have used the terms in much the same way as when they were originally introduced during the 1950's to indicate that point at which additional increments of lime no longer provided an appreciable increase in plastic limit (22, 25, 26). In other cases it was thought to be that point at which the resulting floc size became maximum (27). It has further been associated with being a pH point just short of the required to cause breakdown of silica and alumina complexes and clay minerals (28).

Further studies of these various approaches to define exactly where amelioration effects are maximized and where strength is not appreciably increased, appear to be of questionable value since it became apparent that minor strength increases were obtained simply by reduction of a soils plasticity, and that amelioration effects themselves tended to change during long term curing.

Eades and Grim (28), after considerable review of the lime fixation phenomenon, devised a pH dependent test to evaluate the "point" experimentally. The authors were careful to avoid the use of the terms "fixation" or "retention" in their procedure description, preferring to explain that theirs was a method for determining the lime requirement for "stabilization" of a soil. (See Figure 3)

Because of the considerable difference of opinion as to the definition and significance of this point, the authors elected to define their lime requirement point as that percentage of lime "consumed" by the soil during the first hour following mixing, that is, the percentage at which a pH versus lime curve for the soil became asymptotic. The resulting percentage might have been somewhat larger than that amount which would have resulted through the use of Ho and Handy's (26) "lime retention" approach, since sizable strength increases were observed by Eades and Grim (28) after long-term curing. This might be attributed, at least partially, to the fact that the pH method was based on an



Figure 3 . Lime determination for soil stabilization using data of Eades and Grim<sup>28</sup>

asymptotic point at a pH of 12.3 to 12.4. A soil-lime mixture tends to continue increasing in strength until pH values are well below 11.0 since a pH of this value is sufficient to dissolve silica and form calcium silicate hydrates.

More recent studies (19) to clarify the soil-lime mechanism have resulted in several interesting interpretations:

1. Lime is absorbed physically and at a rapid rate onto most clays from the mix. This absorption removes both calcium and hydroxyl ions from solution concurrently, disagreeing with the calcium crowding concept suggested by the aforementioned Iowa State investigators.

2. The total amounts of lime to be absorbed in these tests tends to conform with values of lime fixation points and to sorption of a little more than a monomolecular layer of calcium hydroxide on the external surface of the clay.

Extension of the above discussion led to the proposal that instantaneous formation of tetracalcium aluminate hydrates  $(C_{4}AH_{13})$  at edge to face points cause an almost immediate flocculation of particles and thereby increase the effective particle size, as well as establishing a structure which impedes swelling or shrinkage activity which might result from variations in the soils moisture content (19).

# Long term effects

That portion of the reaction between lime and soil which primarily takes place after the first hour or so of

cure has been called "long term," "delayed," or "pozzolanic" reaction. It consists, primarily of pozzolanic changes to the mineral although Diamond and Kinter feel that even this is insufficient as a complete explanation for what occurs during this period. It consists of the slow formation of poorly crystalized hydrated calcium silicates and aluminates which serve to strongly cement constituent particles together. Whether the reaction is between liberated silica and lime or between the lime and the clay surface appears to be a bone of contention among different authors (19). Experimental evidence indicates that the predominant reactions take place between the clay mineral fraction and that little or no reaction takes place with other major constituents of the soil such as quartz (29).

A table of reaction products compiled from numerous tests is presented in Table 1. The types and amounts of product are apparently dependent on the mineralogy of the constituents, the type of lime, the temperature of cure and the amounts of water present. These investigations were conducted on standard clays at high lime and water contents and often at high temperature curing in order to assure adequate amounts of reaction product for x-ray diffraction testing.

The long term reaction mechanism is greatly enhanced by high soil moisture content, high curing humidity and high temperature curing. Laboratory control of these factors

Chaminan Manu	Curing Time								
oC	1 Day	3 Days	7 Days	14 Days	28 Days 56 Day				
5	A?,G	A?G,II	A?,G,II	A?,G,II	A?,G,II	A,G,II			
23	A,G	A?G,II	A?,G,II	A?,G,II	A?,G,II	A,G,II			
чo	A?,G	A?G,II	A?,G,II	A?G,II	A?,G,II	A,G,II			
50	A?,G	A?G,II	A,Ga,IIa	A,Ga,IIa	A,Ga,Ia,IIa	N.D.			
60	A?,Ga,IIa	A,G,II	A,Ga,IIa	A,Ga,Ia,IIa	N.D.	N.D.			

ТΑ	BLE	1
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# SUMMARY OF CRYSTALLINE PRODUCTS OBSERVED FROM X-RAY ANALYSIS<sup>a</sup>

 $\begin{array}{ccc} A - -C_{l_{4}}A^{\dagger}H_{n} & C - -Ca0 \\ G - -CSH(gel) \\ I - -CSH(I) \\ II - -CSH(II) \end{array} Tobermorites \begin{array}{c} A^{\dagger} - -Al_{2}O_{3} \\ H - -H_{2}O \\ S - -SiO_{2} \end{array}$ 

1.10

?--Probable

a--Probable Lattice substitution

N.D.--Not determined

<sup>a</sup>After Ruff and Ho (31).

N 00 speeds the rate of strength increase and improves the crystallinity and even changes the type of cementing products developed.

High temperature curing might be expected to result in soil strength modification beyond that which might be expected in the field (28, 30, 31, 32). Relationships between laboratory cured and ambient cured specimens indicate that this is indeed the case (32). (See Figure 4)

#### Strength characteristics

Early work in the use of lime as a stabilizing agent was directed towards improving the plastic characteristics of soils. Often it was mixed in small quantities with portland cement to improve the "workability" of the mixture during construction. As it became obvious that higher percentages of lime were capable of producing increased strengths in the soil, research and development activities in this area of stabilization were undertaken on an ever widening scale.

Many methods were used to measure the strength development in soils as a result of lime stabilization. These included bearing (23), unconfined compression (35), penetration (34), triaxial (33) and durability testing (35). All but the last of these methods indicated that strength of clay-lime mixtures increased substantially in periods of weeks and that the increases in strength continued for months, even years, provided sufficient amounts of lime were



Figure 4 . Effect of high temperature and field curing on lime stabilized soil, (after Anday).

used and moisture and temperature conditions were maintained within certain limits.

Durability testing is usually based on the ability of a soil to maintain its strength characteristics through alternate cycles of wet-dry or freeze-thaw. Perhaps the least desirable attribute of the lime stabilized clay soil is its relative inability to retain strength after being subjected to wet-dry or freeze-thaw cycles. The best solution for this shortcoming has apparently been to refrain from using lime in situations where such weathering conditions might be expected to occur.

Difficulties in predicting the eventual strengths of soil-lime mixtures have been experienced as a result of the very slow curing process during which most of the strength increase is developed. A method which has proven somewhat fruitful is the application of high temperature curing (31, 32). Although this method has met with reasonable success in predicting eventual "long term" strengths in the laboratory, perturbations caused by field conditions often result in substantial deviations of developed strengths from those predicted. Despite these difficulties, laboratory results, when interpreted correctly, provide at least an approximate idea of the eventual strength of the stabilized soil.

The fact that a predominantly clay shale falls into the soil category may introduce another difficulty. The very nature of shales, even when treated in the laboratory

tends to impair the efficient distribution of lime throughout the soil mass. Bonds between particles in "clusters" or "domains" often are sufficiently strong to keep water-borne lime, either in molecular or ionic (Ca<sup>++</sup> and OH<sup>-</sup>) form, away from the total available surface area. The exact manner in which this inability to effect complete coverage of constituent particle surfaces affects the soil mass depends on the ability of the original bonds to maintain themselves in their new environment.

Some consideration might be given here to the theory of lime migration, that ability of lime to travel through a soil mass for long distances over extended periods of time and thereby modify that soil significantly (20). Despite some success claimed for this theory, lime migration seems significantly dependent on the ability of water to carry the lime through the soil medium, that is, its permeability. Because of the extremely low permeabilities of most shales, lime migration effects are probably minimal, if experienced at all.

# Consolidation characteristics

The effects of lime modification on the consolidation response of cohesive soils have not been widely investigated in the past. It seems likely that such a situation occurred because studies of the modification of other properties were considered more desirable at the time.

Some work of an exploratory nature was conducted by Laguros (36) during the middle sixties. This work was performed in an attempt to measure the effect of consolidation on compacted raw and stabilized (and remolded) soils at their as-compacted and saturated moisture content. The results of this program indicated that the amount of consolidation settlement was significantly reduced by lime modification and that composite e-log P curves for several stabilizing agents including lime indicated that stabilization is equivalent to compaction, densification or preconsolidation. (See Figure 5).

Individual time-deformation curves for increased pressures also indicated that settlement times were significantly reduced by treatment and that  $C_V$  (coefficient of consolidation) values, accordingly, increased. It was further proposed that the  $C_V$  values appeared to vary, percentage wise, as the activity of the clay, especially in the case of kaolinite and montmorillonite.

The above facts might be considered in light of previously discussed theory. An increase in the compressive strength of a lime stabilized soil involves the creation of a fabric or structural framework within the soil mass, suggesting that increased elastic response to loading or unloading pressures might be expected in an ideally limemodified soil case. The modulus of elasticity, E, for limesoil mixtures were found to be increased 5 to 14 times by



Figure 5a. Effect of stabilization on clay soils, (after Laguros).



Figure 5b. Effect of stabilization on clay soils, (after Laguros).

Thompson (37) which lends support to such a premise. The extent to which elastic response is effected is, of course, dependent on the amount of lime added to the soil. In the case of clays, the amount should be sufficient to cover all particles and thereby secure the clay samples from contact with moisture. In the case of shales which are partially cemented, the fact that clusters or domains of clay particles might be wholly or partially covered by a monomolecular layer of lime could cause a considerable reduction in consolidation settlement without modifying the surface of each individual particle.

The extent to which soil consolidation response might be affected by lime modification is dependent on several factors including the type of clay mineral, the amount of clay mineral, and the amount of lime used.

The work of Laguros (36) with predominantly clay soils showed that the stabilization of three of these clays, montmorillonite, illite and daolinite, with 6 percent lime increased  $C_V$  values from 15 to 167 percent in 28 days ambient curing depending on the type of clay mineral used.

In his paper, Kondner (38) implies that a need exists for developing a "composite parameter" for clay mixtures to enable the evaluation of their consolidation response. Further work on developing this parameter has not apparently been attempted.

Further complications arise from the presence of other-than-clay components in commonly encountered soils, including shales.

#### Summary

There appears to exist a need to investigate the effects of lime stabilization on shales of the intermediate (non rock-like, non soil-like) variety, particularly shales of this type which are common to the State of Oklahoma. Tests to investigate the modifications which take place in the unconfined compressive strength and consolidation response of shales were selected as a means of observing these effects because of the direct applicability of the properties involved to highway construction. Strength testing includes: determination of maximum increases in unconfined compressive strengths, the effect of clay fraction amount and mineralogy on strength development and the relationship of strength development to other modified properties including soil pH and reaction product increase.

Because of lack of available information at the present time, consolidation testing involved the observation of trends associated with consolidation response modifications resulting from lime stabilization, rather than a laborious and complicated explanation of the results.

# CHAPTER III

## SELECTION OF MATERIALS

#### Shales

A total of twenty typical Oklahoma shales obtained through the Oklahoma Department of Highways were available for use by the investigator. A portion of these shales had been used in connection with weathering studies conducted by the Oklahoma University Research Institute and they were, because of their different response to weathering, ideal for the purposes of this study. Disturbed samples were collected in accordance with AASHO Standard Method T 26-64 and placed into plastic lined cloth bags for transportation and storage.

The samples selected fell into the soil-like and partially rock-like or indurated categories as described by Underwood (3). They were initially excavated in apparently rock-like condition and broke down on weathering in a relatively short time (several days to several months), into their constituent particles, or at least very nearly so.

A number of standard tests were run on these soils to note variation in their physiochemical and engineering properties. These included:

1. Grain size determinations .-- Grain size determinations were made in accordance with the AASHO Standard Method of Mechanical Analysis of Soils, T 88-57, with one exception: additional specimens of each sample were subjected to ultrasonic vibration in a Westinghouse "Mini Magnapak" during the last eight hours of the 24 hour soak in a dispersant solution. Two distribution curves, one for standard analysis and a second for ultrasonic-enhanced analysis, are shown for samples 12 and 24, the least and most "weatherable" specimens. (See Figures 6 and 7). These curves indicated that the samples selected were close to completely broken down to their constituent particles as a result of the selected soil preparation procedures, hence showing a tendency for low weathering resistance. Clay fraction amounts were determined from the grain size distribution curves for both the less-than 5 and less-than 2 micron ranges and are presented in Table 2.

2. Clay mineralogy.--Clay mineral determinations were obtained employing x-ray diffraction techniques. Both bulk powder and sedimented slide specimens were used and standard tests for mineral differentation employed (glycolation, heating, etc.). DTA and chemical analyses using x-ray fluorescence were performed to support x-ray diffraction findings.

3. Atterberg Limits.--Liquid limit, plastic limit and plasticity index determinations were made in accordance with AASHO methods T 89-60 and T 90-61.



SIEVE NUMBER OR HYDROMETER READING TIME

Figure 6 . Comparison of gradation characteristics for sample 12.



Figure 7. Comparison of gradation characteristics for sample 20.

41

In addition, the classification of shales according to three soil classification methods (Textural, Unified and AASHO) was made utilizing applicable property values determined in the foregoing tests.

The total number of twenty shales was reduced to six representative samples. The primary bases of selection were variations in soil properties as follows:

- plasticity as determined by Atterberg Limits tests,
- predominant clay minerals noted in x-ray diffraction analyses, and
- clay fraction amounts as determined in hydrometer analyses.

Results of physiochemical and engineering property determinations made in the selected shales are furnished in Tables 2 and 3. Site locations are depicted in Figure 8.

A short geologic description of the shale samples selected is as follows:

Sample 12 (Washita).--This shale was formed during the Upper Cretaceous period and lies in the Gulf Coast Plain, a belt of gently dipping geologic units extending across the southern portion of McCurtain County, and covering folding and faulting units of the Ouachita Mountains. The Washita Unit consists mainly of shaley clays, blue to black and weathering to light grey or yellow (40). Thickness of

TABLE 2

PROPERTIES OF SHALES	PRC	PERTIES	0F	SHALES
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S	ample		Tex	tural Com	<i>K</i> )		Property			
		01 7 -1		0114	· (7)	(7) em	;	Physical		Che
No.	Location (County)	Okianoma Geologic Unit	kianoma         Sand         Sift         Clay         Clay           eologic         (0.074-         (0.005mm))         (<0.002mm)           Unit         (2-0.074mm)         0.005mm)         (<0.002mm)		Sand Silt Clay Cla (0.074- 0.074mm) 0.005mm) (<0.005mm) (<0.00		LL <sup>b</sup> (%)	PI <sup>C</sup> (%)	S.G. <sup>d</sup> (g/cc)	C.E.C (meq/1
12	McCurtin	Washita	0.5	15.5	84.0	68.0	83	38	2.62	38
13	McCurtin	Washita	6.0	34.0	60.0	48.0	43	23	2.73	20
18	Greer	Flower- pot	0.0	23.5	76.5	56.0	Կ1	11	2.78	21
20	Tilman	Claypool	20.0	41.0	60.0	35.0	40	17	2.78	21
22	Carter	Springer- Goddard	0.5	17.5	82.0	60.0	64	29	2.68	30
24	McIntosh	Senora	1.5	60.5	38.0	24.0	29	6	2.73	20

<sup>a</sup>AASHO Method T 88-57.
<sup>b</sup>AASHO Method T 89-60.
<sup>c</sup>AASHO Method T 90-61.
<sup>d</sup>AASHO Method T 100-60.
<sup>e</sup>Cation exchange capacity determined by continuous titration method (56).
<sup>f</sup>Glass electrode method using 10g shale in 50cc distilled water.
<sup>g</sup>X-ray diffraction analysis.
<sup>h</sup>Triangular chart, U.S. Bureau of Public Roads.
<sup>i</sup>TM No. 3-357 Method, Corps of Engineers.
<sup>j</sup>AASHO Method M145-49.
<sup>k</sup>M=montmorillonite.
<sup>l</sup>Q=quartz.
<sup>m</sup>I=illite.
<sup>n</sup>F=feldspar.

TABLE	2
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PROPERTIES OF SHALES

()	8)			Prop	erty									
	01		Phys:	ical	Chemica	al	Ducidan	New	Classification		Classifi	ation		
m)	(<0.002mm)	LL <sup>b</sup> (%)	LL <sup>b</sup> PI <sup>C</sup> S.G. <sup>d</sup> (%) (%) (g/cc)		C.E.C. <sup>e</sup> (meq/100g)	C.E.C. <sup>e</sup> meq/100g) pH <sup>f</sup>		Clay Minerals <sup>g</sup>	Textural	<sup>h</sup> Unified <sup>i</sup>	AASHO j			
	68.0	83	38	2.62	38	7.8	Mk	Ql	Clay	MH	A-7-5(20)			
	48.0	43	23	2.73	20	5.1	М	Q	Clay	CL	A-7-6(14)			
	56.0	41	11	2.78	21	8.1	Im	Q,F <sup>n</sup>	Clay	ML	A-7-6(9)			
	35.0	40	17	2.78	21	9.4	I	Q,F	Clay	CL	A-6(11)			
	60.0	64	29	2.68	30	8.4	М	Q	Clay	MH	A-7-5(20)			
	24.0	29	6	2.73	20	7.6	I	Q,F	Silty- Clay	ML	A-4(8)			

titration method (56). stilled water.

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Com-			Sam	ple					
ponent	12	13	18	20	22	24			
Si0 <sub>2</sub>	57.14	65.88	56.71	59.06	53.94	50.31			
A1203	17.85	16.19	16.33	16.86	19.49	17.75			
Fe203	9.72	11.40	9.36	13.00	13.62	14.86			
Mg O	2.04	0.84	5.73	2.58	1.93	1.43			
Na <sub>2</sub> 0	0.18	0.08	0.68	0.81	0.18	0.27			
К <sub>2</sub> 0	3.21	0.76	4.88	2.40	1.42	3.50			
Ca0	3.30	0.91	0.50	0.43	1.11	2.20			
Ti0 <sub>2</sub>	0.88	1.26	0.97	1.15	1.52	1.02			
P205	0.15	0.07	0.10	0.08	0.11	0.26			
S	0.02	0.06	0.10	0.01	0.04	0.07			
Cl	0.01	0.01	0.02	0.03	0.01	0.01			
MnO	0.35	0.02	0.06	0.03	0.04	1.12			
L.O.I.ª		4.68	5.63	5.20	9.57	9.62			
Total	102.62	102.16	101.07	101.64	102.98	102.42			

TABLE 3 X-RAY FLUORESCENCE ANALYSIS DATA

a<sub>Loss</sub> on Ignition.



Figure 8 . Locations of sampling sites in Oklahoma.

the unit at the sample location is about 100 to 150 feet. The color of the sample was grey-black.

Sample 13 (Washita).--This sample is similar to sample 12 except for apparent "varving" with red silt and sand layers (40).

Sample 18 (Flowerpot).--This Middle Permian shale, lies on the northern edge of the Hollis Basin in Greer County. The unit overlies thick shale sequences and is gently rolling in nature. The type of vegetation is typical of rich salt or gypsum soils. Flowerpot shales are either reddish brown or greenish grey in color (41). The sample selected was of the latter color and was located at the northern outcrop.

Sample 20 (Claypool).--This shale was deposited in the Lower Permian Period. It lies well into the Hollis Basin in southeastern Tillman County. The unit is primarily found as an outcrop of maroon shales, 1/4 to 1 mile wide along existing stream beds (41). The sample site was in such an outcrop.

Sample 22 (Springer-Goddard).--Sample 22 is a Pennsylvanian shale which lies in Ardmore County between the Arbuckle Mountains and the Criner Hills Uplift. The beds are highly folded with steep dips up to 60° quite common. Valleys are underlain by thick shale sequences with highly plastic olive to dark grey platy shale outcrops. The

Springer-Goddard unit forms broad flat prairie valleys (39). The sample selected was dark grey in color.

Sample 24 (Senora).--Formed during the Middle Pennsylvanian Period, the sample was taken from the upper portion of 500 foot outcrops in western McIntosh County. It is usually found interlayered with sandy and silty shales, and is dark grey to black in color (41).

Pressures exerted on these shales during their geologic lifetimes were responsible, in great part, for their basic characters. The histories of the shales go back as far as 240 million years and peak overburden heights occurred at one or more points during this period. In the case of Washita shales -- 12 and 13 -- these peak heights were probably something less than 1000 feet. Flowerpot and Claypool shales--18 and 20--in southwestern Oklahoma had considerably higher overburden--in the range of 2500 to 3500 feet. The Senora and Springer-Goddard shales--22 and 24--underwent at least one and possibly two loading cycles. For example, the Senora unit attained a loading height of around 7000 feet during its first 170 million years. This eroded down to approximately 1000 feet and then a second loading cycle took place, increasing the overburden height to around 2500 feet during the past 70 million years (42).

#### Lime

A large variety of commercial hydrated limes was available to the investigator for use in this research. The

criterion for its selection was simply that it should be typical of limes normally used in the stabilization of Oklahoma soils. The type eventually selected was furnished by the St. Clair Lime Company, Oklahoma City. Its chemical analysis is shown in Table 4. The lime was placed in large containers and covered to reduce possible carbonation or other contamination during storage.

# TABLE 4

CHEMICAL ANALYSIS FOR LIME<sup>a</sup>

95-98%	•	•	U	۰	•	o	٥	٠	•	a0	C
0.20-0.35%	•	0	•	•	•	•	•	٠	•	lg0	Ν
.003%	o	•	•	•	ů	0	٥	υ	) <sub>3</sub>	'e2(	F
0.05-0.20%		0	•	•	•	o	a	o	<sup>о</sup> з	.120	A
0.05-0.15%	•	3	٠	υ	U	۰	•	•	2	10 <sub>2</sub>	S
Trace	٥	•	•	•	۰	•	0	•	o	0	S
Trace	•	•	0	٠	•	•	0	٥	วี	20	F
0.5-1.0%	•	υ	1.	ioi	it	gn:	IĮ	on	5 (	oss	I

<sup>a</sup>Furnished by St. Clair Lime Company, Oklahoma City, Oklahoma.

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#### CHAPTER IV

# RESEARCH PROCEDURE

# Strength and Associated Testing

#### Preliminary investigation

Three of the selected shales were subjected to preliminary study. This included investigation on the methodology of procedures which could be used in evaluating the developed unconfined compressive strengths of lime modified shales. The result of this "pilot" study was the formulation of the procedure described below.

# Sample preparation

Sample preparation was accomplished in accordance with AASHO procedure T 87-57 with the exception of crushing and grinding which was accomplished by using the Hewitt Soil Grinder. (See Figure 10). This equipment is advertized to accomplish soil breakdown without crushing individual particles. Grain-size distribution curves obtained by using this method of preparation and others obtained by using the mortar and pestle method of preparation were compared and, for the samples involved, seemed to support this claim. This sample preparation method necessarily implies that the



Figure 9. Testing procedure for lime stablized shales.



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Figure 10 . Hewitt Soil Grinder

testing procedures selected will involve use of "remolded" rather than undisturbed specimens. It should be noted therefore, that all references to raw or lime modified shales assume a remolded specimen condition.

Two times the amount of test-required material was placed into plastic bags and then tied. The bags were then placed into two-gallon ice cream cylinders to maintain uniform moisture contents. Routine hygroscopic moisture determinations were performed every week or two during the testing period as a check of the adequacy of the storage method.

#### Lime\_requirement\_determination

The method of Eades and Grim (28) in which the point of sufficient lime for amelioration was defined as the amount required to raise the pH of the mix to approximately 12.4 (or to a constant level approaching that value) was used. The percentages of lime for stabilization were selected to provide this minimum amount plus an additional excess amount to assure measurable pozzolanic strengthening. (See Figure 3).

### Moisture requirement determination

Optimum moisture contents and maximum dry density values were obtained for the soil-lime combinations noted above. These determinations were made using the method recommended by ASTM (43) for use with the Harvard Miniature Compaction Apparatus.

#### Batch preparation

The total amount of shale-lime mix was sufficient to prepare thirty 1.315 inch diameter x 2.816 inch high specimens for each lime percentage and for each sample shale. This allowed three specimens to be used for each cure time Shale-lime combinations at each percentage of lime period. were dry mixed in a specially hooded Hobart Mixer for two minutes. (See Figure 11). The hood was designed to prevent the loss of materials through spilling or suspension in air. The batch was then manually spoon mixed for three additional minutes. Individual 125 gram portions were then removed and placed into individual quart sized stainless steel bowls. The appropriate amount of distilled water was added after using predetermined optimum moisture content and hygroscopic moisture figures in calculating the moisture requirement. This was then mixed for a period of time sufficient to yield a thoroughly blended material.

#### Specimen compaction

The mixture was placed into a Harvard Miniature Compaction mold in five lifts. The compaction tamper used was preset at 20 pounds of load, and was applied 25 times per lift. The specimen was then trimmed, removed from the mold and sent into the wrapping operation.



Figure 11. Hobart Mixer with hood.

#### Curing preparation

In order to prepare the specimens for curing, each was immediately wrapped in a sheet of plastic "saran wrap" and taped with plastic tape to assure a tight seal. Another covering consisting of aluminum foil sealed with masking tape was then applied. The test specimen was then taped and placed into one of two curing containers, both designed to maintain close to 100 percent humidity conditions during curing. (See Figures 12 and 13).

Short-term cure specimens were placed into individual jars which had been specially fitted with porcelain stands below which five milliliters of distilled water had been added to provide a constantly available humidity source. Lids were tightly screwed onto the bottles to prevent loss of Each set of three long-term cure specimens (3) moisture. days-6 months) was placed into a glass tray with aluminum riser. Eighty milliliters of distilled water was left on the bottom of the trays to provide not less than 90 percent relative humidity for curing. After the specimens had been placed in the tray, they were covered with a soaked paper towel. A Butyl rubber sealent was put on the top edge of the glass tray, and a thin gauge aluminum cover was fitted over the tray top. A double strip of duct tape was placed along the cover-tray seam and the tray tagged. The exterior was dried and the entire container weighed to provide a reference to check excessive moisture loss. (See Figure 13).



Figure 12. Single specimen curing preparation stages: a. compacted, b. wrapped with plastic, c. wrapped with aluminum foil, and d. sealed in curing bottle.



Figure 13. Multiple specimen curing preparation stages for high temperature curing: a. aluminum riser tray, b. glass tray, c. water and riser, d. wrapped specimens, e. soaked paper towel, f. butyl rubber sealant, aluminum cover and duct tape seal.
# <u>High temperature and</u> <u>ambient curing</u>

High speed, high temperature curing was adopted for this study both as an expedient to provide data on a time basis and as a means of predicting eventual strengths at the end of time periods considerably beyond the study program length.

The application of this method of strength prediction by several highway departments in the United States seems based on the amount of success that it has met with since its introduction several years ago. The method appears quite accurate in forecasting eventual, though ideal, strengths of clay-lime mixtures and has been successful to a somewhat lesser degree in determining specific cure period strengths, that is, relating short term cure strengths to strength at a particular time in the normal cure cycle (32). It is noteworthy that practical applications are further restricted by lack of field control of temperature and moisture conditions.

The cure temperature of  $140 \pm 5^{\circ}F$  was adopted because of its use in prior experiments and its ability to yield relatively consistent results in them. Although use of this temperature as a maximizing one for all clay minerals is subject to some question (31), the differences in strengths obtained from employing different temperatures do not seem to warrant this latter approach. Further, support for using a single temperature might be based on the fact that two or

more different clay minerals are present in the shales selected for study.

A total of 70 percent of the specimens prepared were cured in the  $140^{\circ}F$  oven while the remaining 30 percent were cured under ambient conditions (72 ±  $3^{\circ}F$ ). Periodic checks were made to assure the maintenance of cure temperatures.

The selection of appropriate cure periods for strength development of the compacted shale lime specimens involved the following considerations:

1. Standard engineering tests involving strength determinations of stabilized soils generally require cure periods of seven days or 28 days. Since strength increases as a result of lime stabilization in the field takes place over even longer periods, cure periods of 90 days and six months are often used.

2. Amelioration effects are considered completed after one hour of curing has been accomplished. Testing conducted during preliminary investigations indicated that one hour curing strengths using ambient curing did not differ appreciably from those obtained during high temperature curing. This apparently is due to the fact that one hour is sufficient time only for temperature stabilization within the specimen and so chemical attack by the excess lime on the clay minerals is not enhanced to an appreciable extent.

3. The time required for moderate percentages (3-6 percent) of lime to attain maximum developed strength

appeared, during preliminary testing, to occur within the first 28 days of high temperature cure.

4. Similarly, the time required for low percentages (1-3 percent) of lime to reach the maximum strength point appeared to occur within the first six months of ambient cure.

5. Critical relationship points between high temperature and 28 day ambient cured strength vs time curves for preliminary tests seemed to occur within the 12 to 36 hour high temperature cure period.

6. The curves noted in 5 above tended to be similar in shape.

7. The time limit for the primary phase of the research program was six months.

After consideration was given to the above, cure times were selected as follows:

High Temperature Cure Periods:

1 hour 3 hours 9 hours 24 hours 3 days 9 days 28 days Ambient Cure Periods: 9 days 28 days 90 days

180 days

The three hour, nine hour and three day periods were chosen in order to "fill in" curve points. These periods

form an approximately geometric progression based on the factor 3, thereby giving relatively uniform spacing in a semilogarithmic plot such as is normally used in showing the strength-time relationship.

## <u>Unconfined Compressive</u> <u>Strength testing</u>

Specimens were removed from the curing oven or from ambient curing shelves upon completion of the appropriate cure period, removed from their containers and unwrapped.

Unconfined Compressive Strength (UCS) testing to failure was then performed on a Soiltest Compression Testing machine, model AP-170B, with a load capacity of 10,000 pounds. (See Figure 14).

Dial readings were recorded and later converted to strength terms and three-specimen averages determined.

Unconfined compressive strength vs time charts were prepared using these average values to show the progress of strength with increased cure time for each treated sample. (See Figures 18 to 23).

# Post-UCS specimen preparation

A portion (approximately 20 gm) of the failed specimen was used for moisture content determination. The figures obtained were used to compare the actual moisture content with that designed for the test and to detect moisture content variations during the course of curing. Actual moisture contents fell in the range of +5.8 to -3.8 percent of the





Figure 14. Compression strength testing device.

design moisture content value. For each batch, the maximum moisture content variation for any one specimen at the end of the curing period was ± 1 percentage point. A one-third portion of each failed specimen was placed into a vacuum dessicator for forty-eight hours. The dessicant used was an indicating variety of anhydrous calcium sulfate, commercially produced under the commercial name, "Drierite." Carbon dioxide removal during the drying operation was accomplished by using another commercial indicating crystal product, "Mallicosorb."

Each of the dessicant-dried specimens was carefully ground in a concrete grinder and the material placed into two separate glass bottles, one portion to be used in determining the mixture pH at the end of the cure period, and the other to be used in detecting variations which took place in mineralogy within the sample at its particular lime content and cure period.

#### pH Testing

Tests performed in this phase of the study to determine soil pH conformed, in general, to one described by M. L. Jackson (44). This method consists of using a 5 to 1 ratio of distilled water to soil, on a weight basis, and a one hour agitated mix prior to testing. The pH meter used was a Sargent Model LS, glass electrode type. In order to minimize deviations caused by testing each specimen as it became available after the drying operation, the dry

specimens were stored in sealed bottles until all specimens for one sample and percentage had been assembled. All pH values were then determined, recorded and used in the preparation of pH-time graphs. (See Figures 29 to 34).

#### X-ray Diffraction analysis

Specimens for x-ray study were stored in a manner similar to those mentioned above, to reduce the effect of further reaction between the clay and lime, until all specimens of each sample had been assembled. Powder pack patterns were then run on a Siemen's x-ray Diffraction Unit through the three to sixty degree (20) range. The powders were then subjected to solvation with ethylene glycol,  $C_2H_6O_2$ , and additional patterns run. A control specimen of the raw shale was prepared and tested in a similar manner. Results of this testing are shown in Figures 35 to 40.

# Consolidation Testing

#### Preliminary investigation

As in the strength testing phase, the establishment of a formal consolidation test procedure as described in the following paragraphs was based on the results of preliminary investigational testing.

#### Sample preparation

Sample preparation for the Consolidation Test was accomplished in accordance with AASHO method T 87-57, again, with the exception of using the Hewitt Soil Grinder instead

of a rubber mortar and pestle. The shales used were taken from the same sample bags as for strength testing and it was assumed that the composition of the materials used was the same for both phases. The storage of samples between its dry preparation and the specimen preparation was accomplished in the same manner as for strength testing.

#### Lime requirement determination

The determination of lime requirements incorporated consideration of the results of strength testing and was performed in two ways:

Method A.--It was noted during strength testing that ambient cured 28-day unconfined compressive strength was reached in 12 to 36 hours of high temperature cure. In general, the higher the percentage of lime, the faster the 28-day UCS was reached. For the sake of convenience, more than for any other reason, the consolidation test specimens were prepared at that percentage of lime which most closely yielded 28-day ambient strength in approximately 24 hours of high temperature curing for each sample.

Method B.--The implications of the above described method of lime requirement selection, was satisfactory for studying the effects of lime on the consolidation properties of each shale. However, it was far from satisfactory when it became necessary to compare results of testing between different samples. This function was better served by selecting a uniform percentage of lime for all six shale

samples. An intermediate value of 4.0 percent was chosen for this purpose.

### Moisture content determination

Moisture content requirements for specimen preparation were determined in a similar manner to that used for strength testing, and involved that amount of moisture required to obtain maximum dry density as determined by using the Harvard Miniature Compaction Apparatus.

#### Batch preparation

A total of 450 grams of the prepared sample was used in each specimen preparation. In the case of lime modified specimens, this amount was mixed, dry, with an appropriate amount of the stabilizing agent in a Hobart Mixer for two minutes, and by hand for three minutes. The required amount of water was then added and the entire mixture hand blended until it appeared to have a uniform consistency.

#### Compaction and trimming

The mixture was placed into a Proctor mold and compacted with 25 blows from a 5.5 pound rammer dropped 12 inches. This conforms to the preparation of a single level of soil as normally performed in AASHO Standard Method T 99-61.

The specimen was then removed from the mold by means of a hydraulically operated extruder and placed on a manually operated specimen trimmer for insertion into a 2.5 inch diameter by 1 inch high brass floating ring. (See Figure 15). After insertion, the specimen was trimmed to make its exposed surfaces flush with the face of the ring.

# Curing

The lime-shale specimen and ring was then wrapped with one layer of plastic wrap and one layer of aluminum foil in a similar manner to strength specimens. The specimen was then placed into a stainless steel bowl which contained a 1/2 inch riser block. An amount of distilled water, sufficient to reach a little below the top of the block was added to provide a humidity supporting water surface during curing. The bowl was then wrapped with aluminum foil, a tight seal being assured, and placed into a 140°F oven for the appropriate time to obtain an approximation of 28-day ambient cure strength.

# <u>Consolidation loading and</u> <u>unloading</u>

After curing, or, in the case of non-modified shales, after trimming, the specimen was weighed and placed between two large porous stones and soaked in distilled water for 24 hours to assure saturation. After the 24 hours soak, the specimen was trimmed, weighed and placed into the consolidometer for testing.

Equipment used in the consolidation test included a levermatic consolidation apparatus, a 2.5 inch diameter, 1 inch high floating ring, a floating ring consolidometer, a



Figure 15. Trimming apparatus for consolidation specimen preparation.

dial indicator to record displacements on loading and a weight set including weights giving loading pressures of 1/8, 1/4, 1/2, 1, 2, 4, and 8 tons per square foot. (See Figure 16).

An operational modification was made to the consolidation test in order to minimize the loss of soil between the bottom porous stone and the floating fing. This loss had been experienced during preliminary testing on the non-modified specimens, as a result of trying to maintain constant saturation by flooding the specimen and porous stones. The scheme shown in Figure 17 was devised as a result and utilized the water retention capability of the porous stone to maintain saturation. Some additional care was exercised to maintain the two separate water surfaces throughout the course of the test.

An additional modification was made to selecting a consolidation period for the load increments rather than using a uniform test period for each load. A deformationrate-dependent method was devised. A record of deformation rates was maintained and the loading period was terminated when the deformation rate fell below .00001 in/min. This was done for several reasons:

1. Consolidation testing and computations are based on determining the effects of primary consolidation. Often, too long loading periods involve considerable secondary consolidation, particularly in the case of more stable



Figure 16 . Levermatic consolidation testing device.



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Figure 17 . Method of maintaining saturation of consolidation specimens.

soils (45). In this case, primary consolidation was considered to be effectively complete when the deflection rate decreased to about .00001 in/min.

2. Preliminary tests indicated that primary consolidation in the case of stabilized shales often occurred in a matter of minutes.

3. Preliminary tests also indicated that primary consolidation in the case of non-modified but partially cemented shales often occurred in a matter of a few hours.

For these reasons it was felt that loading periods of 24 hours usually recommended for cohesive soils could be unnecessarily long, and seriously affect the number of tests available in the study. A deformation-rate-dependent method was therefore considered to be more appropriate.

Complete records of both loading and unloading data were maintained throughout this phase of the test because the modifications to conventional methods noted above.

Specimens prepared using lime contents as determined in Method A, were used to compare the response of treated and untreated samples to static loading.

Specimens prepared using lime contents as determined in Method B, above, were used to compare the response of similarly treated specimens to static loading. The load intensities used were selected to correspond with loading ranges which might be expected for normal highway and highway structure construction. These loadings were: 1/16, 1/4, 1/2, 1, 2, 4 and 8 tons per square foot.

Deformation,  $t_{90}$ ,  $C_v$  values and void ratio versus log pressure curves were prepared from the recorded data and calculations and are presented in Figures 43 to 88 and Tables 10 to 18.

Consolidation responses under the respective consolidation pressures are computed in terms of the coefficient of consolidation  $C_v$  in  $10^{-4}$  cm<sup>2</sup>/sec, using the conventional square root of time fitting method:

$$C_v = \frac{0.848 \text{ H}^2}{\text{t}_{90}}$$

in which H represents the longest drainage path in centimeters and  $t_{90}$  the elapsed time in seconds for 90% consolidation (46). Taking conversion factors and recording methods into account, the expression may be modified into the following form for use in this study:

$$C_{v} = \frac{0.3^{42} (d_1 + d_2)^2}{t_{90}}$$

in which  $d_1$  represents the thickness of the specimen at start of the load increment and  $d_2$  represents the thickness at the end of the load increment.

#### CHAPTER V

# DISCUSSION OF FINDINGS

# General

As noted in Chapter II, different research approaches were used in the two phases of this study:

1. Strength modification of lime stabilized shales was investigated in light of its time development rather than simply its evaluation at a specific curing time. The latter approach has been investigated widely and has become quite standardized whereas the former has been the object of very little study.

2. Conventional consolidation testing methods have been applied so little to stabilized soils that their applicability as evaluation tools in this area has not been established yet. For this reason a comparison of consolidation response characteristics between raw and stabilized shales were selected as a prime subject for research.

Test data were collated into Figures 18 through 40, and Tables 5 through 9 to facilitate interpretation of findings.

As an additional convenience, the terms "montmorillonite shales" or "montmorillonite samples" will be used to denote samples whose clay fractions consist predominantly of that mineral. A similar designation will be made for samples whose clay fraction consists predominantly of illite.

# Strength Development Through Lime Stabilization

The term "Strength Development" as used in the remainder of this study refers to the gradual increase in the unconfined compressive strength (UCS) of shale-lime specimens with increases in curing time. The "strength development histories" of particular shale-lime combinations are depicted in Figures 18 through 23. Separate curves are presented in these figures for high temperature and ambient temperature curing. Additional data discussed in this section of the chapter are furnished in Tables 5 through 9 and Figures 2<sup>4</sup> through 40.

#### Maximum strengths

A tabulation of the maximum strengths developed for samples with varying amounts of lime has been provided in Table 5. Whenever the strengths at the final test curing period had not reached an apparently maximum value, as indicated by a leveling of the appropriate strength development curve, the tabulated value was annotated with a plus (+) sign. The values in Table 5 indicated that the greatest



Figure 18. Effect of method of curing on strength of shale 12 stabilized with lime.



CURING TIME, HOURS

Figure 19. Effect of method of curing on strength of shale 13stabilized with lime.



Figure 20. Effect of method of curing on strength of shale 18 stabilized with lime.



CURING TIME, HOURS

Figure 21. Effect of method of curing on strength of shale 20stabilized with lime.



Figure 22. Effect of method of curing on strength of shale 22 stabilized with lime.



Figure 23. Effect of method of curing on strength of shale 24 stabilized with lime.

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final strengths attained were in illite shales. This appeared true both in total strengths attained and in percentage increase from raw shale strengths.

This was even more clearly evident when an additional tabulation (Table 6) was prepared showing the maximum strengths attained by montmorillonite and illite shales subjected to treatment with nearly equal amounts of lime (4.0 to 4.5 percent). The values in this table supported the observation that for long term high temperature curing illite samples tended to be more significantly modified than montmorillonite samples. The same tendency was at least implied in the case of ambient cured specimens, despite the fact that, based on the six month test observation point, the "maximum" strength value of illite shale 24 seemed slightly out of line with those of the other samples. If one observes, however, that all three of the illite shales had not yet reached their maximum strengths at the final ambient cure test time, this incongruity is not as significant as it first appears.

The ability of illite shales to attain these higher maximum strengths seems to be based primarily on the higher "cement"<sup>\*</sup> to surface area ratios obtainable in illite shales for approximately the same amount of lime-clay reaction product.

<sup>\*&</sup>quot;Cement" refers to any cementitious products formed by the reaction of lime and the clay mineral surface.

# TABLE 5

MAXIMUM STRENGTH DATA

	·	High Temp. Cure		Ambient Cure		
Sample No	% Lime, (%)	Maximum UCS, (psi)	Increase, <sup>a</sup> (%)	Maximum UCS, (psi)	Increase, <sup>a</sup> (%)	
12	0	50				
	3	120	140	107	114	
	6	230	360	189	278	
13	0	37				
	2.1	1 58	327	142	281	
	4.2	245	562	203	450	
18	0	39				
	2	155	297	100+	1 50+	
	Դ	325+	333+	235+	502+	
20	0	34				
	1.5	1 50	341			
	2	205	502			
	3	280+	724+	258+	660+	
	4.5	440+	1194+	340+	900+	
22	0	31				
	3	128	313			
	λ <sup>†</sup>	173	458			
	6	221	615	214	590	
	8	375	1110+	196	532	
24	0	28				
	1.5	75	168			
	3	133+	395+	89+	218+	
	4.5	300+	971+	148+	430+	

<sup>a</sup>Increase in UCS over UCS of Raw Shale.

	Sample No.	Lime Content, (%)	High Temp Cure		Ambient Cure	
			Max. UCS, (psi)	Increase, (%)	Max. UCS, (psi)	Increase, (%)
Montmorillonite	12	4,5	160	220	_a	_a
bliates	13	4.2	245	562	203	450
	22	4.0	175	458	_a	_a
Illite Shales	18	4.0	325	733	235+	504+
	20	4.5	440+	1194+	340+	998+
	24	4.5	300+	971+	1 <sup>1</sup> +8+	430+

# TABLE 6

MAXIMUM STRENGTH DATA FOR SHALES WITH 4.0 TO 4.5 PERCENT LIME

<sup>a</sup>Strength testing at these percentages of lime was accomplished only during the preliminary phase and for high temperature cure specimens.

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### Effect of clay mineralogy

Figure 24 compares the recorded strength development histories of two shales of differing mineralogy, both of which had been treated with three percent lime. Shale 22. whose clay fraction was predominantly montmorillonite appeared to react early with the stabilizing agent immediately beginning gradual but uniform strength increase rate until it reacted a maximum point of approximately 128 psi after three days of high temperature curing. Shale 20, whose clay fraction was predominantly illite, appeared, on the other hand, to delay any substantial reaction until approximately 9 hours of curing and then began a higher rate of strength increase up to 3 days. From this point the strength continued to increase at a lesser rate up to and probably beyond the final 28 day test point when the unconfined compression strength was approximately 280 psi.

It was apparent that, although all of the tested montmorillonite shales developed lower maximum strengths than the illite shales, they were inclined to attain these strengths during earlier stages of curing.

The slower strength increase in illites might be attributed to two factors:

1. Type of reaction product formed.--Various studies (30, 49, 50, 51), have indicated that lime-clay reaction products vary from one clay mineral to another and from one curing temperature to another, but that they are



98

CURING TIME, HOURS

Figure 24. Effect of clay mineral on strength development of lime stabilized shales 20 and 22.

basically either calcium silicate or calcium aluminate hydrates and cementitious in nature. Jambor (52) has studied "lime-pozzolano" pastes and found that their strengths are affected by the types and microstructures of the developed calcium silicate hydrates. Metcalf (48) has reported that the lime reactions which cause strength in lime-clay mixtures are not the same for all clay minerals. Also, from these studies it can not be inferred that the reaction rates are necessarily the same for two different minerals at the same curing temperature.

2. Amount of reaction product formed. -- A somewhat more acceptable explanation of the observed results might be based on the fact that the average particle size of a typical montmorillonite is considerably smaller than that of a typical illite. This would result in giving the former a substantially greater surface area per unit weight of soil mass for reaction with lime. The statistical probability of any single reaction occurring is, of course, greater for that surface area which is larger. Given a certain number of possible reaction points therefore, it would be expected that the total number of reactions per unit time for the larger surface area would be higher, which implies that the rate of reaction would be faster, as would be the formation of the reaction product. The formation of higher amounts of reaction products causes higher early strength development. Eventually, however, the total number of reactions

for both surface areas would approach the same number. This greater cement (reaction product) to surface area ratio would at this point, favor the lower surface area soil, re-sulting in a higher maximum strength.

#### Effect of clay amount

An interesting phenomenon is noted in the case of illite and montmorillonite shales differing in clay fraction amounts. This is depicted in Figure 25 which shows two montmorillonite shales, 13 and 22, which had been modified using four percent lime. It can be seen that, despite the fact that sample 22, which contained the larger clay fraction amount, tended to increase its strength (to 178 psi) earlier, sample 13 gradually achieved a higher final strength (280 psi).

Several possible causes might be considered in this situation:

1. The larger clay fraction amount provides more clay surface with which the lime might react, hence an earlier using up of the Ca(OH)<sub>2</sub> in the mixture.

2. Although the larger clay fraction amount provides a greater surface area, this area itself tends to reduce the concentration of developed cements within the entire sample mass by decreasing the cement to surface area ratios.

3. The lower total surface area available in the lower clay content shale, though it slows the complete



CURING TIME, HOURS Figure 25. Effect of amount of clay on strength development of lime stabilized shales 13 and 22.

reaction of lime with the clay minerals, eventually increases the effective cement to surface area ratio and thereby provides a more complete strengthening framework to the shale mass.

The same tendency is implied by comparing the strengths of lower clay amount shales 20 and 2<sup>4</sup> with shale 18 (see Table 6). In this case, however, final values of strength had not yet been attained by the end of the final test cure period.

#### Effect of lime amount

Figures 26 and 27 show the strength development curves for montmorillonite and illite shales with different percentages of lime.

It is noteworthy that the strengths developed were not significantly different for differing amounts of lime until an apparent "break point" in the strength development curves were reached. This point appeared to occur at a time when the lower percentage lime-shale reached a nearly maximum strength as indicated by a leveling of the curve.

This reflects the fact that lime-shale reaction products were being produced at essentially the same rate, irrespective of lime amount, up to a point when insufficient excess lime was left with which the clay could react.



Figure 26. Effect of amount of lime on strength development of stabilized shale 13.



Figure 27. Effect of amount of lime on strength development of stabilized shale 18.

# Effect of high temperature curing

Despite considerable past work with various soillime combinations in the area of high temperature curing, there seemed to exist some need for confirming the adequacy of the high temperature curing method to predict the eventual strength development of shales consisting of various clay fraction amounts and mineralogical combinations. It was decided that two methods would be employed to make this confirmation: (1) pH comparison and (2) 28 day strength comparison.

1. The first method involved comparing the pH values of the stabilized shale samples at specific strength values using both the high temperature and ambient curing methods. This procedure was based on the fact that pH may be used as an indicator of lime utilization in the mixture. A lowering of pH value is associated with a decrease in the amount of lime available for reaction with the clay mineral and a corresponding increase in the reaction product available to effect strength increase. The progressive decrease in pH with curing is depicted in Figures 28 to 33.

Table 7 compares the pH values for specimens cured 90 days under ambient conditions with calculated pH values for the specimens of the same strengths cured at 140°F. The latter pH was obtained in the following manner: (See Figure 34) One of the strength development curves for a specific sample at a given lime content was entered and its




Figure28. pH comparison method for shale 18.



CURING TIME, HOURS

Figure 29. Effect of method of curing on the pH of shale12 stabilized with lime.



Figure 30. Effect of method of curing on the pH of shale 13 stabilized with lime.



Figure 31. Effect of method of curing on the pH of shale 18 stabilized with lime.



Figure 32. Effect of method of curing on the pH of shale 20 stabilized with lime.



Figure 33. Effect of method of curing on the pH of shale 22 stabilized with lime.

Time	90 Day UCS-	Hi Temp Cure Time for		pH	
Content (%)	Ambient, (psi)	Same UCS, (hrs)	Ambient	Hi Temp	Deviation from Amb.
6.0	190	40 40	11.50	11.85	+0.35
3.0	107	72	10.95	11.15	+0.20
4.2	204	68	11.80	11.80	0.00
2.1	143	60.	11.10	11.10	0.00
4.0	138	60	11.35	11.75	+0.40
2.0	92	53	11.10	11.10	0.00
4.5	233	47	11.80	12.20	+0,40
3.0	220	72	11.70	11.55	-0.15
6.0	202	72	11.60	11.45	-0.15
8.0	190	62	12.10	12.15	+0.05
4.5	145	105	11.60	11.85	+0.25
3.0	105	100	10.75	11.35	+0.60
	Lime Content (%) 6.0 3.0 4.2 2.1 4.0 2.0 4.5 3.0 6.0 8.0 4.5 3.0 6.0 8.0 4.5 3.0	Lime UCS- Content (%) UCS- Ambient, (psi) 6.0 190 3.0 107 4.2 204 2.1 143 4.0 138 2.0 92 4.5 233 3.0 220 6.0 202 8.0 190 4.5 145 3.0 105	Lime Content $(\%)$ 90 Day UCS- Ambient, $(psi)$ Hi Temp Cure Time for Same UCS, $(hrs)$ $6.0$ 19040 $3.0$ 10772 $4.2$ 20468 $2.1$ 14360 $4.0$ 13860 $2.0$ 9253 $4.5$ 23347 $3.0$ 22072 $6.0$ 20272 $8.0$ 19062 $4.5$ 145105 $3.0$ 105100	$\begin{array}{c c c c c c c c c c c c c c c c c c c $	$\begin{array}{c c c c c c c c c c c c c c c c c c c $

TABLE 7

pH COMPARISON DATA FOR 90 DAY AMBIENT STRENGTH VALUES



Figure 34. Effect of method of curing on the pH of shale 24 stabilized with lime.

90 day ambient strength point was determined. a) A horizontal projection of this point was made to the high temperature curve for the same shale at the same lime content. b) The time reading for this projected point was recorded and c) the chart showing the variation of pH with time for the same mixture was entered for the recorded time. This yielded a calculated pH value for a specimen at a high temperature cure strength equal to its 90 day ambient strength. d) This pH value was then compared to the pH of the 90 day ambient specimen (see Table 7). The high temperature curing seemed to yield somewhat higher pH values than ambient curing for the same unconfined compressive strength. An explanation for this might be tied onto the results of studies conducted at the Iowa State (30, 31, 49) during the past decade which have noted that the use of high temperature curing produces reaction products of higher crystallinity or of slightly different chemical composition. If, as might be expected, the higher crystallinity of reaction products results in either greater cementing effectiveness or cement strength, the effect of a given amount of lime would be to produce higher mixture strengths when the mixture is cured at higher temperatures. It would also follow that, in comparing strength development curves for ambient and high temperature cured samples, a given strength for a high temperature cured sample should be associated with a higher pH value than the same strength for an ambient cured sample. It is noteworthy

that the slightly higher pH values for high temperature cured specimens were associated with small deviations which fell well within those which might be expected for the equipment and test methods employed.

A slightly different approach to the pH comparison was used by attempting to determine the status of lime utilization (pH) at or near maximum developed strength employing the two methods of curing. Table 8 shows the results of this comparison. The average pH value obtained for the high temperature cured specimens at or near maximum strength was 10.9 and for the ambient cured specimens was 11.3. The difference was primarily due to the fact that, for ambient curing, the illite shales had obviously not reached their maximum strengths by the time the final curing period was This conforms with similar predictions of Eades complete. and Grim which infer that strengthening of soil-lime mixtures continues for a long period and is not essentially complete until the pH is reduced to below approximately 11.0 (28). It was evident, however, that pH values were very nearly the same in cases where both ambient and high temperature strength development curves for the same shale-lime combination had apparently reached a leveling off point. (See Table 8 for shales 12, 13 and 22)

2. The second method of confirming the adequacy of high temperature curing as a predictive tool of ambient strength was previously referred to as a 28 day strength

## TABLE 8

<u> </u>	Lime	pH		
No.	(%)	Hi Temp	Ambient	
12	3.0	11.1	11.1	
13	2.1	10.9	11.1	
18	2,0	10.7	11.2 <sup>a</sup>	
20	3.0	10.8	11.7 <sup>a</sup>	
22	6.0	11.4	11.6	
24	3.0	10.3	11.2 <sup>a</sup>	

## pH VALUES AT MAXIMUM DEVELOPED STRENGTH VALUES

<sup>a</sup>Maximum strength apparently not reached within cure period.

comparison. It was based on the previously discussed findings of preliminary strength testing which indicated that 24 hour high temperature  $(140^{\circ}F)$  curing strengths compared favorably with actual 28 day strengths. Using the 24 hour high temperature cured strengths as predictive values, therefore, a comparison was made with actual 28 day ambient strengths. (See Table 9). Predicted strength values for the twelve shale-lime combinations fell within the range of +27.2 to -31.2 percent of the actual ambient values, the mean deviation from ambient values being +5.7 percent. This would tend to indicate that the selected high temperature curing period was adequate for predicting 28 day ambient strengths.

#### X-Ray diffraction

X-ray diffractograms for raw and stabilized shales are presented as Figures 35 through 40.

For the raw shales, these diffractograms indicated that clay minerals had typical d-spacings corresponding to  $2\Theta$  angles (53). Also, the clay mineral information thus obtained is closely related to other engineering properties as evident from the data in Table 2.

A study of the patterns for lime stabilized shales failed to reveal the presence of measurable amounts of reaction products as it was expected in view of previous studies (26, 29, 31). The patterns of stabilized shales cured at 140°F for 28 days indicated little apparent

# TABLE 9

	Timo	UCS (psi) for		
Sample No.	Content, (%)	24 Hour High Temp	28 Day Ambient	Deviation (%)
12	3	85	90	- 5.5
	6	143	114	25.4
13	2.1	124	100	24.0
	4.2	108	90	20.0
18	2	63	72	-12.5
	¥-	74	80	- 7.5
20	3	137	103	27.2
	4.5	116	107	8.4
22	6	121	105	15.2
	8	110	99	11.1
24	3	55	59	- 6.8
	4.5	57	83	-31.2

## COMPARISON OF PREDICTED AND ACTUAL UNCONFINED COMPRESSIVE STRENGTH VALUES



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9 10 4.5 15 20 3.5 5 8 4.0 6 7 T 4.2% LIME, HYDRATED 0% LIME, HYDRATED 4.2% LIME, GLYCOLATED 0% LIME, GLYCOLATED 24 22 20 18 16 14 26 12 10 8 6 4 Degrees of Arc -  $2\theta$ 

INTERPLANAR SPACING - d, IN ANGSTROMS

Figure 36. X-ray diffraction patterns for sample 13.



Figure 37 . X-ray diffraction patterns for sample 18.



Figure 38. X-ray diffraction patterns for sample 20.



Figure 39. X-ray diffraction patterns for sample 22.

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Figure 40 . X-ray diffraction patterns for sample 24.

modification had taken place in the mineralogical properties of the clay fraction. The expandability of montmorillonite clays by ethylene glycol was relatively unchanged by stabilization. This paradox may be due to a number of factors:

1. The shales are made up of sand, silt and clay fractions. Most previous researches were conducted on pure or nearly pure clays.

2. The amounts of lime used are very small compared to the entire shale mass.

3. The crystallinity of reaction products is low, especially at ambient temperature and humidity conditions.

4. The pulverization method used to prepare the samples for x-ray diffraction analysis caused mechanical breakdown of many of the cementing bonds formed during stabilization or further breakdown of particle clusters. Such mechanical action allows clay minerals to react in a normal manner with water or other expanding liquids such as ethylene glycol. Because the reaction with lime is a surficial phenomenon, the major portion of the minerals present in the clays are not modified through reaction with lime, and since the affected surfaces are greatly broken down by the pulverization process, the overall effect of lime is masked by the normal response of the clay minerals to x-ray diffraction analysis.

#### <u>Selecting cure periods for</u> <u>strength evaluation</u>

One practical consideration is suggested by the results obtained in the strength development phase of this study -- in the case of lime stabilized illite shales, 28 day ambient curing or 24 hour high temperature curing seemed insufficient to obtain any sizable reaction between lime and the clay mineral. For example, (see Figure 20) in the case of sample 18, four percent lime gives less than 22 percent of the final strength after 24 hours of high temperature curing, while in the case of sample 22 (see Figure 22) the same amount of lime gives over 93 percent of the final strength under the same conditions of curing. The obvious hypothesis which arises from these observations is that 28 days of curing might be inadequate for the purpose of determining the eventual strength (or consolidation) response of lime stabilized illitic clays or shales. The problem, however, seems to lie in the fact that there was sufficient excess lime present in the illite-lime mixture after the selected cure period to eventually cause substantially greater strength development. This excess apparently did not coincide with the percentage of lime selected in the case of the montmorillonite shale. Had the percentage selected for the latter shale been greater, a similar situation could have resulted. In any event, these results suggest the need for careful selection of cure periods in the case of lime stabilized shales, especially when the amount of lime

used is intended to provide sufficient excess amounts to cause substantial follow-on modifications to the mixture.

#### Change in Consolidation Response through Lime Stabilization

#### <u>Compression-decompression</u> <u>deformations</u>

Tables 10 and 11 presents the total deformation values obtained from raw and stabilized shales during the loading (compression) and unloading (decompression) portions of the consolidation test.

In general, total deformations of saturated raw specimens, were within predictable ranges. Samples of higher clay fraction amounts and predominantly montmorillonite mineralogy exhibited the greater tendency to deform under load and to rebound after load removal. Samples which had lower amounts of either montmorillonite or illite fell in the middle value range. (See Table 10)

Consolidation testing of samples stabilized using method A indicated that substantial reductions in total deformations occurred. Compression deformations of 8.4 to 23.2 percent and decompression deformations of 11.6 to 36.3 percent of those for raw shales were recorded. In the case of samples stabilized using this method, however, a complete comparison between improvements wrought by lime was somewhat obscured by the fact that different amounts of lime were used on different samples. (See Table 11)

## TABLE 10

Sample No.	Total Compression Deformation, (inches)	Total Decompression Deformation, (inches)	Decompression- Compression Ratio (DCR)
12	0.2291	0.0999	•436
13	0.1625	0.0725	•447
18	0.1296	0.0555	.428
20	0.1772	0.0759	.428
22	0.2228	0.0823	•369
24	0.1183	0.0438	•371

### RAW SHALE CONSOLIDATION TEST DATA

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Sample No.	Lime Content, (%)	Defl.(in.)	Defl. Stab. Defl. Raw	Decc Defl.(in.)	Defl. Stab.,(%) Defl. Raw	DCR
12	4.0	0.0214	9.8	0.0151	15.3	.705
	6.0	0.0245	10.7	0.0141	14.1	• 576
13	4.0	0.0141	8.7	0.0097	13.3	.688
18	4.0	0.0301	23.2	0.0127	22.8	.422
20	4.0	0.0270	15.7	0.0149	19.6	• 536
	4.5	0.0284	16.0	0.0119	15.7	•419
22	4.0	0.0202	9.1	0.0129	15.7	.639
	8.0	0.0187	8.4	0.0095	11.6	• 508
24	3.0	0.0231	19.5	0.0159	36.3	.689
	4.0	0.0204	18.9	0.0112	23.6	• 549

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# TABLE 11

## LIME STABILIZED SHALE CONSOLIDATION TEST DATA

Samples stabilized by method B, using uniform amounts (4.0 percent) of lime, underwent total compression deformation amounts of 9.1 to 23.2 percent and decompression deformations of 15.3 to 25.6 percent of those for raw shales.

It appeared significant that the decompression deformation to compression deformation ratios, hereinafter noted as DCR, for the shales tended to increase substantially with stabilization, implying that the stabilized shale recovers a large portion of its original shape, and thus has an increased elasticity. Table 11 and Figure 41 show how these ratios varied from shale to shale and with variations in lime content.

Whereas the values of DCR for raw shales did not seem to bear any specific relationship to the mineralogy of the clay fraction, those obtained for lime stabilized shales apparently did. Figure 41 shows that DCR values of montmorillonite shale number 22 begin to increase with increasing lime content up to about 0.70 at 4.2 percent lime and then to decrease beyond that point. A similar situation exists for montmorillonite shale 12 in which the maximum DCR of approximately 0.64 occurs at a lime content of 4.2 percent. Illite shales have maximum DCR values at lower lime contents. Shale 20 has a maximum DCR of 0.60 at 2.8 percent and shale 24 has a maximum DCR of 0.69 at 2.7 percent lime. The optimum lime content for maximum DCR appears

therefore to be dependent on the surface area available in the shale mass. This maximum DCR value appears to signify that lime content which would result in a maximum increase in the elasticity of the soil for the curing time involved. The optimum lime content point might be expected to vary with the amount of surface area available for reaction with lime. The lower DCR values obtained for lime contents below the optimum lime content were apparently caused by the lower amounts of reaction products which are formed, and the lower DCR values obtained for lime contents higher than the optimum lime content apparently reflect the presence of excessive, unused lime.

The results depicted in Figure 41 suggest that a family of curves may be developed for various curing periods, wherein optimum DCR values will be occurring at increasingly higher lime contents as the reaction products are progressively formed.

# Deformation during incremental loading and unloading

Prior to stabilization, raw shales exhibited various deformation paths through incremental loading. Some showed nearly uniform deformations for most of the loading sequences, while others varied more or less unpredictably. The total deformation for each load, as measured just prior to the addition of a new load, is presented in Figures 42 to 47.



Figure 41. Effect of lime on recovery of consolidation deformations.







Figure 43. Effect of consolidation load increments on deformation (d) of raw shale 13.



Figure 44 . Effect of consolidation load increments on deformation (d) of raw shale 18.



Figure 45 . Effect of consolidation load increments on deformation (d) of raw shale 20.







Figure 47. Effect of consolidation load increments on deformation (d) of raw shale 24.

In general, raw illite shales displayed a more uniform deformation pattern during successive load increments whereas montmorillonite shales showed a tendency towards appreciable increase in deformation with load increase. This seemed particularly evident in the 1/2 to 4 Tsf range. (For loading pressures up to 1/2 Tsf, the tendency of the raw shales seems to be more of "seating" or otherwise accommodation of the loading head and porous stones to the specimen.) The consolidation test in this case involved a remolded shale, with a more random particle (or particle cluster) orientation than would exist in the shale in its natural environment. The compaction effort used in preparing the remolded specimen at maximum density actually produces a soil structure somewhere between that associated with the "cardhouse structure" and that of the "fully oriented" condition (54). The addition of saturation water and concurrent application of initial loading pressures causes an internal breakdown of the structures of the shale mass thus yielding a higher degree of particle orientation.

Step-by-step unloading produced similar results (see Figures 48 to 53) in decompression values for raw shales, although there was some increase in deformation values as loading decreased.

Deformations during incremental loading and unloading in the case of stabilized shales, as shown in Figures 54 to 65, increased or decreased more uniformly than for the



Figure 48 . Effect of consolidation load decrements on deformation (d) of raw shale 12.



Figure 49. Effect of consolidation load decrements on deformation (d) of raw shale 13.



Figure 50. Effect of consolidation load decrements on deformation (d) of raw shale 18.



Figure 51 . Effect of consolidation load decrements on deformation (d) of raw shale 20.



Figure 52 . Effect of consolidation load decrements of deformation (d) of raw shale 22.



Figure 53 . Effect of consolidation load decrements of deformation (d) of raw shale 24.



Figure 54. Effect of consolidation load increments on deformation (d) of lime stabilized. shale 12.



Figure 55. Effect of consolidation load increments on deformation (d) of lime stabilized. shale 13.



Figure 56. Effect of consolidation load increments on deformation (d) of lime stabilized. shale 18.



Figure 57. Effect of consolidation load increments on deformation (d) of limestabilized shale 20.



Figure 58. Effect of consolidation load increments on deformation (d) of lime stabilized shale 22.



Figure 59. Effect of consolidation load increments on deformation (d) of lime stabilized shale 24.



Figure 60 . Effect of consolidation load decrements on deformation (d) of lime stabilized shale 12.



Figure 61 . Effect of consolidation load decrements on deformation (d) of lime stabilized shale 13.



Figure 62 . Effect of consolidation load decrements on deformation (d) of lime stabilized shale 18.


Figure 63 . Effect of consolidation load decrements on deformation (d) of lime stabilized shale 20.



Figure  $_{64}$  . Effect of consolidation load decrements on deformation (d) of lime stabilized shale 22.



Figure 65 . Effect of consolidation load decrements on deformation (d) of lime stabilized shale 24.

8 SHALE + 4% LIME E<sub>c</sub> =5050 PSI RAW SHALE AXIAL PRESSURE, Tsf 4 E\_=247 PSI 2 1 E =84 PSI 0 0.1 0.2 0 CONSOLIDATION STRAIN, IN/IN

Figure 66. Consolidation stress-strain relationships for shale 12.

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CONSOLIDATION STRAIN, IN/IN

Figure 67. Consolidation stress-strain relationships for shale 13.





CONSOLIDATION STRAIN, IN/IN

Figure 68 . Consolidation stress-strain relationships for shale 18.



Figure 69. Consolidation stress-strain relationships for shale 20.



Figure 70. Consolidation stress-strain relationships for shale 22.

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Figure 71 . Consolidation stress-strain relationships for shale 24.

## TABLE 12

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Sample No.	Ec <sub>1</sub> (psi)	E <sub>C2</sub> (psi)	E <sub>C3</sub> (psi)
12	84	247	5050
13	167	470	7400
18	139	1390	4620
20	251	1300	3970
22	80	222	5690
21+	145	1350	2580

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### ELASTICITY DATA FOR RAW AND STABILIZED SHALES

raw shales thus suggesting the increase of elasticity in the stabilized shale. The "seating" or "accommodation" response to initial loading increments which took place in the raw shales were not evident in the case of stabilized shales since such response is apparently impeded by the availability of cementing materials at contact points between particles.

#### Elastic properties

Figures 66 to 71 and Table 12 are presented to show the modification to the elastic response of shale specimens which was caused by using four percent lime and 24 hour high temperature curing.

Stress (6) and strain ( $\varepsilon$ ) values were computed from data obtained in the consolidation tests. For this reason, strain values are for specimens which were confined laterally by the floating ring and therefore vary from those normally presented for standard tests (either axial compressive strain (AL/L) or volumetric compressive strain (AV/V). In this case, because of the fixed area of the floating ring, the axial deformations conform with volumetric variations, but the corresponding strain values do not. The strain value is therefore noted as  $_{\rm C}$  and any computation of modulus of elasticity (E) using this strain value is shown similarly as ( $E_{\rm C}$ ) and will be referred to as the consolidation modulus of elasticity.

The modulus of elasticity of any material is represented by the slope of its stress-strain curve. Since a modified method of determining strain is employed, it follows that the value of  $E_c$  might deviate from an E value obtained from using the more standardized tests. It would be desirable, therefore to select a method of computing a meaningful value of  $E_c$  especially in the case of raw shales where the curve slope is difficult to obtain. Two methods for its computation are presented:

1.  $E_{c_1}$  is represented by the straight line portion of the stress-strain curve between the origin and the first two points of the curve. This would appear to be the most conservative value of  $E_c$  for design application.

2.  $E_{c_2}$  is that value obtained by computing the slope of the stress-strain curve at a strain value of 0.1 inches per inch. Such a strain signifies an arbitrarily selected limit above which a soil mass is considered to have failed because of excessive deformation despite its not having failed in shear. The modulus obtained at this point is somewhat higher than that obtained using the previous method but does provide an upper limit  $E_c$  value for the shale in question and allows for a more complete investigation of the variation in the  $E_c$  value which might result from lime stabilization.

Comparison of values obtained from using these two methods are shown in Table 12 and indicate that  $E_{C_2}$  values

were from 2.75 to 10 times greater than  $E_{c_1}$ , somewhat beyond normal factor of safety ranges usually employed in design considerations yet still indicative that use of  $E_{c_2}$  might be reasonable method for calculating a meaningful value of consolidation modulus of elasticity for the raw shales.

 $E_{\rm C}$  values for raw shales whose mineralogy was predominantly montmorillonite, were generally lower than those for illite shales. This was expected because of the greater deformations obtained in the case of montmorillonite shales.

 $E_{\rm C}$  values for raw shales containing montmorillonite clay minerals increased with decreasing clay amount, indicating a loss of the plastic influence of clay and an increasing mobilization of interparticle friction forces as clay amounts decreased.

The effects of lime on the consolidation modulus of elasticity were evident in the following ways:

1. The slopes of stress-strain curves of lime stabilized shales became quite uniform through the entire test range. This is generally accepted as a characteristic of the materials of greater elasticity and a single value of consolidation modulus elasticity,  $E_{C3}$ , was easily calculated.

2.  $E_{C_3}$  values for montmorillonite shales were higher than for illite shales. This substantiated the explanation presented earlier in this study that lime reacts more quickly with the higher amount of surface area provided by

that mineral and thereby improves the resistance to loading of the shale-lime fabric at a faster rate.

The effect of clay fraction amount on the  $\mathrm{E}_{\mathrm{C},\mathrm{S}}$  value was, somewhat more difficult to determine. In the case of montmorillonite samples,  $E_{\mbox{C}_{\mbox{C}_{\mbox{O}}}}$  values increased with decreasing clay fraction amounts while in the case of illite shales,  ${\rm E}_{\rm CQ}$  values decreased with decreasing clay fraction amounts. This might be explained by the fact that, in the case of illite shales, complete utilization of the lime was delayed by coverage of the individual clay particles with a monomolecular layer of lime. Quantities of unused lime could be expected to be greater as the surface area of the soil mass decreases and has an overall weakening effect on the soil mass. It is quite probable that, in the case of montmorillonite shales, the surface area available for reaction was large enough to react with all of the lime during the curing period. It should be noted, however, that with longer curing time, the eventual  ${\rm E}_{\rm c_{\rm l}}$  value for the lime stabilized illite shale could be expected to be greater than that of the montmorillonite shale since the cement to surface area ratio would eventually become greater for the illite shale.

 $E_{c_3}$  values which resulted from lime stabilization varied from approximately 2 to 25 times the  $E_{c_2}$  values obtained for the natural shales. This compares quite favorably with results of Thompson (37) who used more conventional

methods to determine "compression modulii" of soils similar in textural classification to the shales tested herein. Lime contents used in his tests were 5 percent and the curing temperature used was  $120^{\circ}$ F for 24 hours. Although Thompson's E values were considerably higher than the  $E_{c_3}$ values because he used triaxial methods in his investigation, review of his results indicates that increases in compression modulus of elasticity values due to lime stabilization varied from 5 to 14 times those obtained for the natural soil.

#### $t_{90}$ and $C_v$ Values

Values of t<sub>90</sub> and  $C_v$  for each load increment were determined as shown in the example presented in Table 15 and are tabulated in Tables 13 and 14. Several graphical attempts were made to determine if variations in  $C_v$  values for the raw shales were in any way related to their other physicochemical properties.

 $C_v$  values for raw montmorillonite shales were approximately one half of those for illite shales. Nearly all raw samples exhibited either maximum or minimum  $C_v$ 's during the initial increment of loading 1/16 to 1/4 Tsf. This reflected the tendency of specimens to adjust to the testing apparatus, to assume some initial structural stability, or to continue to swell as a result of saturation (see Figures 72 to 77).

## TABLE 13

Sample No.	t <sub>90</sub> Range, (sec)	C <sub>v</sub> Range, (cm <sup>2</sup> /sec)
12	540-5189	0.000204-0.000457
13	1 <i>9</i> 49 <b>-</b> 5134	0.000250-0.000543
18	960 <b>-</b> 2940	0.000452-0.001106
20	960 <b>-</b> 3110	0.000388-0.001398
22	1815-5762	0.000226-0.000563
2 <sup>1</sup> +	1058-2306	0.000577-0.001149

## TIME AND COEFFICIENT OF CONSOLIDATION DATA FOR RAW SHALES

		Rat	w Shale	Stabil:	ized Shale		
Sample No.	Pressure Increment (Tsf)	t <sub>90</sub> (sec)	C <sub>v</sub> (cm <sup>2</sup> /sec)	<sup>t</sup> 90' (sec)	C <sub>v</sub> ' (cm <sup>2</sup> /sec)	$\frac{C_V}{C_V}$	Avg. $\frac{C_{v}}{C_{v}}$
12	1/16-1/4 1/4 -1/2 1/2 -1. 1-2 2-4 4-8	3557 5189 540 2306 2940 2614	0.000367 0.000231 0.002044 0.000437 0.000330 0.000331	15 240 60 41 34 66	0.09110 0.00567 0.02260 0.03290 0.03940 0.01970	248 25 11 75 119 56	88
13	1/16-1/4 1/4 -1/2 1/2 -1 1-2 2-4 4-8	3375 5134 3375 4438 1949 2774	0.000397 0.000350 0.000362 0.000258 0.000543 0.000356	94 15 15 15 15 66	0.01453 0.09070 0.09040 0.09000 0.08940 0.01970	37 258 250 350 164 55	186
18	1/16-1/4 1/4 -1/2 1/2 -1 1-2 2-4 4-8	2940 1500 1325 1109 1109 960	0.000452 0.000843 0.000919 0.001055 0.001007 0.001107	124 197 735 390 224 497	0.01000 0.00691 0.00185 0.00344 0.00600 0.00262	23 8 2 3 6 2	7

## TABLE 14

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TIME AND COEFFICIENT OF CONSOLIDATION DATA FOR RAW AND STABILIZED SHALES

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		Rav	v Shale	Stabili	lzed Shale		
Sample No.	Pressure Increment (Tsf)	t90 (sec)	C <sub>v</sub> (cm <sup>2</sup> /sec)	t <sub>90</sub> ' (sec)	C <sub>V</sub> ' (cm <sup>2</sup> /sec)	$\frac{C_V}{C_V}$	Avg. $\frac{C_{v}}{C_{v}}$
20	1/16-1/4 1/4 -1/2 1/2 -1 1-2 2-4 4-8	960 1270 1009 3110 1949 1949	0.001398 0.001030 0.001260 0.000388 0.000587 0.000500	15 94 54 173 118 183	0.09110 0.01447 0.02510 0.00781 0.01135 0.00711	65 14 20 20 19 14	25
22	1/16-1/4 1/4 -1/2 1/2 -1 1-2 2-4 4-8	5762 3375 2940 1815 1815 3197	0,000226 0.000356 0.000380 0.000563 0.000513 0.000268	25 22 60 28 109 295	0.05450 0.06180 0.02260 0.04825 0.01230 0.00441	241 173 60 86 24 17	100
21+	1/16-1/4 1/4 -1/2 1/2 -1 1-2 2-4 4-8	2306 1109 1058 1239 1296 1413	0.000577 0.001150 0.001172 0.000978 0.000876 0.000769	15 27 49 110 15 177	0.09110 0.05040 0.02770 0.01235 0.08940 0.00734	157 44 24 13 102 10	58

.

TABLE 14--Continued

Applied Pressure (Tsf)	Final Dial Reading (in)	Final Sample Thickness, d (in)	Void Height, H <sub>V</sub> =d-H <sub>S</sub> (in)	Void Ratio, $e=\frac{H_V}{H_S}$	Fitting Time, <sup>t</sup> 90 (sec)	Coefficient of Consolidation, $C_{v} = \frac{\cdot 3^{4} 2 (d_{1} + d_{2})^{2}}{t_{90}}$ $(10^{-4} \text{cm}^{2}/\text{sec})$
1/16	0.0000	1.0000	0.4004	0,668	2206	ב הם
1/4	0.0262	0.9738	0.3742	0.624	2300	2.17
1/2	0.0402	0.9598	0.3602	0.601	1109	11,50
1	0.0584	0.9406	0,3420	0.570	1058	11.70
2	0.0785	0.9215	0.3219	0,537	1239	9.78
<u> </u>	0 1091	0.8909	0 2013	о <u>1</u> 87	1296	8.76
0	0.1091	0.0909	0.2913		1413	7.69
0	0.1103	0.0017	0.2021	0,470	G = 2.73	am/cm3
4	0.1139	0.8861	0.2865	0.478	$u_{s} = 2r/3$	Em\ (сшо
2	0.1090	0.8910	0.2914	0.486	$\mathcal{S}_{W} = 1.0$	gm/cm <sup>3</sup>
1	0.1040	0.8960	0.2964	0.494	A = 4.909	$cm^2$
1/2	0.0978	0.9022	0.3026	0,505	W <sub>S</sub> = 131,9	9 gm
1/4	0.0904	0.9096	0.3100	0.517	$H_s = \frac{W}{M}$	s = .5996 in
1/16	0.0746	0.9254	0.3258	0.543	16.39	G <sub>S</sub> E <sub>W</sub> A

CONSOLIDATION TEST CALCULATIONS FOR RAW SHALE 24

TABLE 15







Figure 74 . Effect of consolidation load increments on  $C_{v}$  of raw shale 18.







Figure 77 . Effect of consolidation load increments on  $C_v$  of raw shale 24.

Figure 78 shows a pattern of  $C_v$  versus liquid limit values for the six sample shales and indicates that the three montmorillonite shales have average  $C_v$  values which decrease with increasing liquid limit. The same seems to be true of the three illite shales. The rate of decrease for both mineralogies appears to be similar though quite small, (approximately  $2.0x10^{-6}$  cm<sup>2</sup>/sec/%) but it is very obvious that the two curves which result are quite distinct from one another.

Figure 79 shows a pattern of  $C_V$  versus clay fraction amount for these same shales. This pattern shows that  $C_V$ values for montmorillonite shales and illite shales also decrease as clay fraction amounts increase. Once again, the pattern for the shales of different mineralogies are clearly separated but of similar slope. Interestingly, this tendency holds true for both the less-than 0.005 mm clay fractions and the less-than 0.002 mm clay fractions.

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Implied in the above two figures is the classical relationship that the liquid limit of a soil increases with its clay fraction amount.

The three relationships noted above indicate that all of the raw shales of the same mineralogy are inclined to act similarly and that their tendency to consolidate on loading tends to increase with increasing clay fraction amount and with increasing liquid limit.



Figure 78.  $C_v$  vs liquid limit curves for raw shales.



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Figure 79. C vs clay fraction amount for raw shales.

A considerable amount of difficulty was encountered in obtaining realistic values of  $t_{90}$  and  $C_v$  for lime stabilized specimens. Classically the  $t_{90}$  value is determined by establishing the straight line portion of the deformation versus square root of time curve (see Figure 80), decreasing its slope by 15 percent and determining the intersection point of this new slope line with the original curve. However, it became difficult to apply this procedure to the stabilized specimens as it is obvious from Figure 81 that the  $t_{90}$  value would have to fall somewhere between the zero reading and the first reading time, in this case, 6.25 seconds. The appropriate estimate of exactly where such a point occurs is questionable at best.

This difficulty is caused by the fact that the classical method of calculating  $t_{90}$  has resulted from extensive observations in consolidation testing of relatively undisturbed cohesive soils. Actually, some doubt can be introduced as to its adequacy if it is observed that quite often  $t_{90}$  values which result are far from representative of the time when 90 percent of consolidation settlement occurs. Some thought was therefore given to adopting the time for 90 percent deformation as the " $t_{90}$ '" values. The appropriateness of this approach coincides with the earlier decision in this study to use the deformation rate as the determinant factor in reaching the completion point of primary consolidation.





Figure 81. Time curve for load increment 1 to 2 Tsf on lime stabilized shale 24.

The entire consolidation records of shales stabilized using method B were reviewed and  $t_{90}$ ' values were determined based on 90 percent of final deformations. The results of this review are shown in Table 1<sup>4</sup> which includes  $C_v$ ' values corresponding to the  $t_{90}$ ' values. These results indicate that  $C_v$ ' values were 2 to 350 times as great as  $C_v$  values for the raw shales. This does not conform closely with the results presented by Laguros (36) who reported increases of only 70 to 167 percent in the case of stabilization of clay soils with 6 percent lime. The disagreement between the two observations accrues from a variety of factors. Two of which seem to be worth discussing:

1. The work by Laguros (36) encompassed C and B horizon soils which contained clays in large amounts but which were not consolidated shales; whereas, in this study, the particulate matter was derived from "shales which were well consolidated.

2. Apparently in the referenced work, the difficulty in determining  $t_{90}$  values was not experienced and therefore, the results of comparing  $C_v$  values with  $C_v$ ' values are questionable, except as noted below:

It can be seen from studying the results shown in Table 1<sup>4</sup> that average  $C_v$ ' values for lime stabilized illite shales varied from  $0.00530 \times 10^{-4} \text{ cm}^2/\text{sec}$  to  $0.04305 \times 10^{-4} \text{ cm}^2/\text{sec}$  and that  $C_v$ ' values for lime stabilized montmorillonite shales varied from  $0.03526 \times 10^{-4} \text{ cm}^2/\text{sec}$  to  $0.06745 \times 10^{-4} \text{ cm}^2/\text{sec}$ 

sec. Also  $C_v$ ' values were an average of 7 to 58 times larger than  $C_v$  values for raw illite shales and  $C_v$ ' values for the three montmorillonite shales were an average of 88 to 186 times the  $C_v$  values of the raw shales.

That stabilized illite shale  $C_v$ ' values were, on the average, lower than the values for montmorillonite shale, was the reverse case of  $C_v$  values obtained for raw shales. These findings reflect the aforementioned fact that montmorillonite shales were more drastically altered than the illite shales by the same amount of lime during the same modified 28 day ambient curing period.

A comparison was made between changes in coefficients of consolidation which resulted from lime stabilization of samples of differing clay mineralogies. This comparison was made on two shales (13 and 24) which had clay fractions and mineralogies very similar to two soils tested by Laguros (36). Data used in this comparison is shown in Table 16. Results indicate that the increase in  $C_v$  values of montmorillonite shale 13 was 321 percent of the increase in the  $C_v$  of illite shale 24. This compared quite closely to a similar comparison made using data from the Laguros study which showed the increase in the  $C_v$  value of the montmorillonite clay soil was 246 percent greater than the increase in the  $C_v$  value of the illite clay soil.

TABLE	1	6
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 $C_v$  increase comparison data

		Clay A	mount	C 1
Sample No.	Clay Mineralogy	0.005 mm (%)	0.002 mm (%)	Avg. $\frac{C_V}{C_V} \ge 100$ (%)
13	Montmorillonite	60	¥8	18600
24	Illite	38	24	. 5800
3C <sup>a</sup>	Montmorillonite	61	¥0	167
20 <sup>a</sup>	Illite	52	34	68

<sup>a</sup>After Laguros (36).

#### Void ratio versus pressure curves

Results of consolidation testing are usually depicted by the void ratio versus pressure curve. Data obtained for both raw and stabilized shales are presented in Figures 82 through 87 and associated calculations and density values are presented in Tables 17 to 19.

#### Preconsolidation

For the most part, the curves for raw shales were typical of high clay content soils with the exception that, normally, a remolded soil will not exhibit a point of preconsolidation load on its curve. However, in all six samples a point of maximum curvature seemed to exist but it was not easily discernable because the slope changes of the curve as a whole were very small. Table 19 shows that these preconsolidation loads varied between 0.4 and 1.65 Tsf for the six shales. The overburden history of the geographic locations from where the shale samples were taken attest to a maximum load of at least:

200 ft of overburden @ 100 pcf =  $\frac{20,000}{2000}$  = 10 Tsf That the preconsolidation load was determinable but that it was considerably lower than the calculated maximum overburden load may be attributed to the remolding effect which is implied in the method of sample preparation used in this testing procedure. This method involved specimens from shales





Figure 83. Compression diagram for raw and lime stabilized shale 13.



Figure 84. Compression diagram for raw and lime stabilized shale 18.



Figure 85. Compression diagram for raw and lime stabilized shale 20.



Figure 86. Compression diagram for raw and lime stabilized shale 22.



Figure 87. Compression diagram for raw and lime stabilized shale 24.

	Density (pcf) at:								Moisture Content (%)			
Gampla	Design	1	Test &	Start	8 1	ſsf	End of	f Test	Des	sign	Satu	rated
No.	Raw St	tab.	Raw	Stab.	Raw	Stab.	Raw	Stab.	Raw	Stab.	Raw	Stab.
12	90.1 8	30,9	66.0	85.0	85.3	87.0	75.7	86.2	28.0	35.5	41.2	36.7
13	111.0 10	04.6	92.9	107.1	110.9	108.8	101.8	108.2	18.1	20.5	26.3	20.4
18	91.8 9	91.5	87.4	97•3	100,4	100.2	94.3	98.6	28.5	29.7	29.9	28.4
20	109.5 10	08.9	103.0	108.5	116.5	111.8	111.5	110.0	18.7	19.6	21.9	20.5
22	92.8 8	36.2	76.3	88.1	98 <b>.</b> 4	90.0	88.8	89.1	27.0	32.0	34.8	31.1
24	112.0 10	07.5	10 <b>1</b> .5	111.0	114.7	113.6	109.8	112.9	18.2	19.2	23.5	19.4

TA.	BLE	17	

MOISTURE-DENSITY VALUES FOR CONSOLIDATION TEST

## TABLE 18

# PRECONSOLIDATION LOADS FOR RAW SHALES

Sample	ə I	No	•			Pı	rec ]		nsolidation ad (Tsf)
12	ø	•	U	•	o	a	σ	o	1.65
13	o	o	n	o	٥	o	0	o	0.80
18	0	•	0	0	0	o	υ	٥	0.90
20	a	o	U	•	U	•	•	a	0.75
22	٥	•	•	U	0	•	•	0	0.40
24	o		0	o	•	•		•	0.75

which, despite being mechanically broken down, had apparently retained within particle clusters or "domains" (55) a portion of the particle orientation and bonding forces originally developed during their formation process. If this were not so, the point of preconsolidation could not be identified at all on the curve.

On the other hand, the corresponding stabilized shale curves did not reveal the presence of a preconsolidation load point. The explanation for the absence of such a point may be found in the fact that the basic structure of the shale had been modified by the addition of lime and the attendant formation of new bonds was sufficiently greater to mask the effects of preconsolidation. Furthermore, the initial densities of stabilized shales used in this portion of the test were considerably higher than those of the raw shales and it is usually difficult to determine the preconsolidation load point when a higher initial density is used.

Table 17 was prepared to show how the densities at the start of consolidation testing varied from the maximum densities, how the densities varied during the course of this testing and how the actual moisture contents varied from the optimum moisture contents as determined by using the Harvard Miniature Compaction apparatus.<sup>a</sup>

<sup>&</sup>lt;sup>a</sup>Optimum moisture contents and maximum densities as obtained by using the Harvard Miniature Compaction Apparatus will henceforth be referred to as "design moisture contents" and "design densities" respectively.

Void ratio and density variations for raw shales

In the case of raw shales, differences between design densities and attained densities at the start of consolidation testing were significantly higher for the montmorillonite shales (16.5 pcf to 24.1 pcf) than for the illite shales (4.4 pcf to 10.5 pcf). (See Table 17). This conforms to the greater swelling ability usually associated with montmorillonite clays.

Also in the case of raw shales, differences between design densities and attained densities at the end of consolidation testing appeared to be closely related to the amount of increase in moisture content between these two points. This is depicted in Figure 88.

End of test densities for raw shales also appeared to reflect the mineralogy of clay components. The density values for all the three montmorillonite shales remained quite far from attaining their design density values by amounts ranging from 4.0 pcf to 14.4 pcf below design, while the illite shales had the end of test densities from 2.2 pcf below to 2.5 pcf above design.

A note should be made in comparing the response between montmorillonite raw shales 12 and 22 as depicted in Figures 82 and 86; these two shales are very similar in mineralogical composition, amount of clay, raw and stabilized strength characteristics and consolidation response in the stabilized condition. The void ratio value at the start of


consolidation testing of sample 12 was approximately 1.40, decreased to 0.85 and rebounded to 1.04 upon completion of testing. Void ratio values for sample 22 at these points were approximately 1.18, 0.70 and 0.88 respectively. An explanation of this variation seems to lie not in their consolidation response as much as in their relative abilities to absorb water during the 24 hour saturation period prior to consolidation testing. The apparently greater ability of shale 12 to absorb water is probably occasioned by its higher cation exchange capacity. Sample 12 had a capacity of approximately 38 meq./100 gms. while sample 22 had a capacity of 30 meq./100 gms. Some support to the effect of the different abilities of the two shales to absorb water is also apparent in their liquid limit and plasticity index values; shale 12 has a liquid limit of 83 and a plasticity index of 38 and shale 22 has a liquid limit of 64 and a plasticity index of 29.

## Void ratio and density variations for stabilized shales

Comparison of the stabilized densities at the start of the consolidation testing with the design densities as shown in Table 17 indicates that, in general, the Proctor method of compaction provided densities slightly higher than the design densities.

After completion of consolidation testing of these stabilized shales, the increases in densities resulting from

the test seemed to be somewhat greater for the illite shales (1.0 pcf to 1.2 pcf) than for the montmorillonite shales (1.3 to 1.9 pcf). This reflects the greater tendency for rebound by the montmorillonite shales previously indicated in the discussion of DCR values.

In the case of stabilized shales, differences between design densities and attained densities at the end of consolidation testing were compared to the amounts of increase in moisture content between the same two points. The comparison shows the beneficial effect of stabilization in terms of the reduced tendency of the stabilized shale to absorb water. It is significant that the moisture content variation after 24 hours of curing and completion of consolidation testing was from 0.9 percent below design to 1.2 percent above design, with a mean variation of 0.1 percent above design.

#### Raw vs stabilized shales

Void ratio versus pressure curves, Figures 82 to 87 and associated Table 19, indicated reductions in raw shale void ratio ranges of 78 to 94 percent at the end of 24 hour high temperature curing using the 4.0 percent design lime content.

A comparison between the void ratio curves for the various shales indicated that the location of the stabilized shale curve with respect to the raw shale curve differed from one shale to another. For example, the stabilized

## TABLE 19

Sample No.	Loading for Raw Shale			Loading for Stabilized Shale			Reduction in e Range During
	1/16 Tsf	8 Tsf	1/16 Tsf <sup>a</sup>	1/16 Tsf	8 Tsf	1/16 Tsf <sup>a</sup>	(%)
12	1,400	.847	1.035	.841	.811	.829	94.4
13	.831	• <b>5</b> 33	.667	• 549	• 509	<b>.</b> 525	86.6
18	• 980	<b>,</b> 723	•833	<u>₀85</u> 2	,807	.838	82.6
20	.607	.404	.505	• 593	. 548	.567	78.0
22	1.184	.699	.879	.896	.857	.882	92.0
24	<u>-</u> 668	.470	<b>.</b> 543	. 564	.532	.550	84.0

# VOID RATIO VARIATIONS DURING CONSOLIDATION TEST FOR RAW AND 4.0% LIME STABILIZED SHALES

<sup>a</sup>Following decompression.

curves for sample 20 seemed to start approximately at the same void ratio, 0.6, as that for the raw shale while for raw sample number 12 the void ratio changes from approximately 1.40 to 0.85 and for stabilized sample number 12 the corresponding change is from 0.84 to 0.81. (Figure 89 depicts the compression-decompression void ratio ranges for all six shales in the raw and stabilized cases.)

Once more, a comparison of the densities at the start of consolidation testing and the density increases which took place during testing might help to explain the apparent difference in the relative locations of these curves.

Table 17 indicates that the initial raw sample densities varied considerably from the design densities. These disparities appeared to be caused by the following two factors:

1. There is a difference in the compactive effort imparted by the Harvard method and the Proctor Compaction method. This difference is more apparent in the case of stabilized shales and is reflected by the fact that the Proctor method imparted a slightly higher density to remolded stabilized specimens.

2. There is a difference, particularly for raw shale, in the response of specimens to the 2<sup>4</sup> hour saturation period prior to testing. This is borne out by the moisture content increases experienced by raw shales which reflect the

higher hydrophyllic character of raw shales, especially montmorillonite, and corresponds to similar finding of many geologists including Grim (10).

Thus, the apparent difference in relative locations of stabilized and raw shale void ratio versus pressure curves is primarily a reflection of the ability of raw shales to absorb water during the 2<sup>4</sup> hour saturation period prior to testing.



Figure 89. Void ratio variations for raw(R) and stabilized(S)shales

### CHAPTER VI

## CONCLUSIONS AND RECOMMENDATIONS

In this study, six Oklahoma shales containing various clay minerals, three predominently montmorillonite and three predominently illite, were subjected to lime stabilization in order to determine the variations in their strength and consolidation properties. On the basis of the data obtained, the following conclusions are presented:

1. Greater maximum strengths were developed in the stabilized illite shales than in the stabilized montmorillonite shales. Using 4.0 to 4.5 percent lime, the strengths of the stabilized montmorillonite shales varied from 160 to 245 psi while the strengths of the stabilized illite shales varied from over 300 to 440 psi.

2. Strength development rates were faster for the montmorillonite shales than for the illite shales treated with the same amount of lime. Maximum strength for the 4.0 to 4.5 percent lime treated montmorillonite shales were reached in approximately 72 hours of 140°F curing whereas maximum strengths for the similarly stabilized illite

shales required over 672 hours of curing time at the same temperature to attain maximum strengths.

3. Montmorillonite shales containing lower clay fraction amounts developed strengths 40 to 50 percent higher than those containing higher clay fraction amounts. Similar strength behavior, but of the order of 20 to 30 percent, was observed for the illite shales.

4. Strength development in the stabilized shales were similar for different lime contents during the initial curing period, beyond which, strength curves diverged, apparently because the shales which had lower original lime contents reduced their excess lime amounts to a level insufficient to maintain a high rate of reaction product formation.

5. High temperature,  $2^{4}$  hour, curing at  $140^{\circ}F$  proved to be a highly effective tool for predicting the 28 day ambient laboratory strength. Predicted strengths were within +27.2 to -31.2 percent of actual values.

6. Values of pH obtained during testing indicated that similar lime utilization was effected for similar strength development under the two systems of curing (high temperature and ambient). For the same ambient, 90 day, and high temperature strengths, the corresponding pH values differed within a range of +0.60 to -0.15.

7. Standard x-ray diffraction analysis techniques could not be used as interpretative tools for determining

the quantity or rate of strength development which took place during curing of lime stabilized shales primarily because of the poor crystallinity of the reaction product and the pulverization method employed in preparing the stabilized shale sample for analysis.

8. Total compression and decompression deformation values for raw shales subjected to consolidation testing reflected the mineralogy and amount of the clay fraction. Raw montmorillonite shales underwent greater deflections than illite shales. Likewise, shales containing greater amounts of clay underwent greater deformation than the shales of the same mineralogy containing smaller amounts of clay. These trends were reversed by treatment with equal amounts, 4 percent, of lime and curing at 140°F for 24 hours.

9. The decompression-compression ratio (DCR) was proposed as a measure of the increased elasticity of a lime treated shale and seemed useful in determining an optimum lime content for maximum increase in elasticity for any selected curing time.

10. From stress-strain curves based on consolidation data, a modified consolidation modulus of elasticity,  $E_c$ , value was introduced to help compare elasticity property variations which resulted from lime stabilization.  $E_c$  values for lime stabilized shales varied from 5 to 25 times the  $E_c$  values of their raw shale counterparts.

11. Standard methods for evaluating  $t_{90}$  and  $C_v$  values proved to be of questionable value in investigating the consolidation response of the stabilized shales. A modified  $t_{00}$ ' value was determined, based on actual 90 percent deformation time. This value was used to determine a modified Cy' value, which could be compared with Cy values of raw The comparison indicated that the coefficient of shales. consolidation values increased from 2 to 350 times as a result of the stabilization with 4.0 percent lime and high temperature curing for 24 hours. Montmorillonite shales benefited more than illite shales using the same amount of lime and curing period. Lime treated montmorillonite shales had average C<sub>v</sub> increases of 88 to 186 times their corresponding raw values while lime treated illite shales had average  $C_v$  increases of 7 to 58 times.

12. Void ratio versus pressure curves indicated reductions in raw shale void ratio ranges of 78 to 94 percent at the end of the 24 hour high temperature curing period using 4.0 percent lime.

13. Pretest densities of raw shales reflected their ability to absorb moisture during the 24 hour saturation period prior to testing while the densities of stabilized shales remained relatively unchanged in spite of this saturation.

14. Density variations during loading from 1/16 to 8 Tsf reflected the mineralogy differences of the clay fraction for both the raw and the stabilized shales. In the case of raw shales, these variations were from 18.0 to 22.1 pcf for montmorillonite shales and 13.0 to 13.5 pcf for illite shales. An opposite trend was observed in the case of stabilized shales and reflected the faster rate of lime modification imparted to montmorillonite shales for the amount of lime and curing period used. Montmorillonite shales had density variations from 1.7 to 2.0 pcf and illite shales had density variations from 2.6 to 3.3 pcf for 4.0 percent lime treatment followed by 24 hours of high temperature curing.

15. The consistently occurring phenomena that (1) montmorillonite shales underwent greater modification in strength and consolidation properties than illite shales; and, (2) that shales of higher clay content underwent greater modification for the same properties than shales of lower clay content, when treated with the same amount of lime and cured for the same 24 hours at  $140^{\circ}$ F, seem based on the relationship of available surface areas between the different shales. For the cure period selected, greater surface area shales allowed higher rates of reaction product formation which resulted in greater strengths or greater resistance to consolidating pressures.

In view of the experience gained in this study, the following recommendations for future study are presented:

1. Investigation of the "fabric" of shales prior to and following lime stabilization by using the scanning electron microscope. Such a means of investigation is not beset with the destructive nature of preparation techniques such as is employed in x-ray diffraction or DTA analysis and is more amenable to particle orientation studies which constitute a basic aspect to the understanding of consolidation and strength properties of clays.

2. Further investigations to evaluate the relationship of high temperature cured laboratory strengths with field results. In conjunction with this, to develop criteria for selecting realistic curing periods based on actual design requirements.

3. Study of a time dependent family of curves to determine the actual role of the decompression-compression ratio (DCR) in increasing the elasticity of soil-lime or shale-lime combinations and to relate its value to other derived elastic properties.

4. Evaluation of relationship between the consolidation modulus of elasticity and modulii derived by presently accepted means.

5. Development of a method for obtaining a more meaningful value for coefficient of consolidation for stabilized soils, one which might be applicable to both raw and stabilized materials and which may be used in standard settlement predictions.

6. Change of the saturation method used prior to consolidation testing. The response of raw shales to this saturation period suggests that, perhaps, more meaningful results might have been obtained if expansion had been prevented by balancing expansion pressures with added loading during saturation. Such a method would have allowed a better comparison of response to added consolidation pressures for raw and stabilized shales beginning at very nearly the same densities.

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