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THE UNIVERSITY OF OKLAHOMA

GRADUATE COLLEGE

ENGINEERING PROPERTIES OF RAW AND ULTRASONIC TREATED OKLAHOMA SHALES

A DISSERTATION

SUBMITTED IN THE GRADUATE FACULTY

in partial fulfillment of the requirements for the

degree of

DOCTOR OF PHILOSOPHY

BY

MANICKAM ANNAMALAI

Norman, Oklahoma

1974

ENGINEERING PROPERTIES OF RAW AND ULTRASONIC TREATED

OKLAHOMA SHALES

APPROVED BY

Charles C. Mathis

for K

Arthur Bernhart

J. Kaguros

DISSERTATION COMMITTEE

ABSTRACT

The effect of ultrasonic treatment on four Oklahoma shales, which were specially selected to present variations in texture and clay mineralogy, was investigated. The engineering properties studied were moisture-density, strength, volume change due to water absorption and consolidation.

It was observed that ultrasonic treatment decreased the density, increased the moisture content, reduced the strength and increased the volume change. Modification of consolidation characteristics included increase in compressibility and decrease in permeability. The extent of modification by ultrasonic treatment appears to closely agree with that by natural weathering; thus suggesting that ultrasonic treatment simulates, to a dependable degree, natural weathering.

As part of this investigation, mathematical relationships were established among moisture-density, volume change, clay content, liquid limit, plasticity index and a new parameter termed "reaction potential" of 24 raw shales which had been naturally weathered to varying degrees. These relationships hold true for ultrasonic treated shales indicating that they can be employed to predict the behavior of the

modified shales and also further ascertaining the simulation phenomenon observed. The composite parameter reaction potential characterizing both type and amount of clay minerals and plasticity index were found to correlate well with the volume change. A second parameter, defined as "disaggregation index" was established for quantification of the effects of ultrasonic treatment and it indicated that the disaggregation mechanism was closely related to the total chemical composition of the shales with CaO resisting it and Fe_2O_3 aiding it.

ACKNOWLEDGMENTS

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CHAPTER I

INTRODUCTION

By virtue of its abundant occurrence as the sedimentary rock in the earth's crust, shale is one of the most widely used construction materials in the state of Oklahoma and elsewhere. The term "shale" is generally used to designate the various argillaceous sediments although at times it describes the subgroup of mudrock or mudstone group. Because of the inconsistency in definition many classification systems for shales have resulted.

The shale classifications are usually not precise enough for engineering purposes. However, the geological classification system which divides shales into the groups of compaction or "soil-like" shales and cemented or "rock-like" shales proves to be a good starting point for classifying shales based on their engineering properties.

Compaction or "soil-like" shales are those which have been consolidated by the weight of the overlying sediments, lack significant amounts of cementing agents, and tend to slake rapidly when subjected to alternate cycles of wetting and drying. Usually these are categorized as

"problem shales." On the other hand, the "rock-like" shales are sufficiently consolidated and well-cemented and generally prove to be construction materials devoid of major weaknesses.

Soil-like shales are more susceptible to weathering than rock-like shales as they lack cementing agents and disintegrate easily. Although the effect of weathering cannot be avoided, a prior knowledge of the end product of weathering and attendant physical properties is very useful for the design and construction of pavements and highway embankments. Thus, some rapid means of obtaining the altered product becomes necessary. In a recent study concluded at the University of Oklahoma, it has been found that the ultrasonic treatment of the shales produce a disaggregated materials and simulate the process of weathering. All the changes brought about by the ultrasonic treatment of shales are not completely known except that the plasticity and gradation characteristics were different for the treated shales. X-ray diffraction patterns for ultrasonic treated samples appeared the same as those for raw shale or untreated shale samples. Based on these observations, the effect of ultrasonic treatment on shale samples is considered to be primarily physico-mechanical.

However, a study of the important engineering properties such as shear strength, volume change and consolidation characteristics of the disaggregated shale materials will help in understanding the effect of ultrasonic treatment on shales and how far this treatment simulated weathering. It

will also indicate the nature and magnitude of changes in the engineering properties to be expected in the weathered materials.

With these objectives in mind, four shales were selected for this study. Three are "soil-like" and one is "rock-like" shale. Specifically the investigation covered the study of

1. The engineering properties, i.e., the index properties, strength, volume change and consolidation characteristics of raw shales;
2. The engineering properties of ultrasonic treated shales;
3. The effect of ultrasonic treatment on the engineering properties; and
4. The disaggregation mechanisms involved in the ultrasonic treatment.

And also attempts have been made to establish some useful correlations among the significant engineering properties of raw shales, relating one property with some of the others, in a manner that these relationships could be employed as predictive tools.

CHAPTER II

REVIEW OF LITERATURE

General

The literature survey on shales indicated, in general, a conspicuous absence of any standardized classification and identification system and the extreme vulnerability of shales to the action of weathering agencies.

Classification of Shales

Underwood (1967) presented a summary of the various attempts by many investigators to classify and identify the shales. He favored the geological classification system, which broadly divided the shales into two groups--"soil-like" shale or the shales which are poorly cemented and are readily disaggregated when slaked with water, and "rock-like" shales or the shales which are well-cemented and resistant to the action of water. Also, based on the engineering properties of various shales such as density, natural water content, volume change, permeability, etc., and their reported performance, he distinguished between the desirable and undesirable shales for use in construction.

Gamble (1971) developed a classification system based on plasticity characteristics and slaking durability of the shales. The slaking durability test measures the resistance of the shale to the action of water and readily classifies shales into soil-like and rock-like shales. Plasticity characteristics are indicative of the engineering properties and performance of the shales. Hence, Gamble's method of classification is, in principle, the same as Underwood's.

Suitability of Shales

The important factors that influence the classification of shales and durability are the type of cementing agents and the amount of clay and clay sized particles. Methods to identify suitable shales, adopted by engineers, included a wide variety of physical tests. Smith, Reidenouer and Distong (1967) used a series of physical tests such as gyratory kneading compaction, wet-dry, Washington degradation, specific gravity and absorption tests. They found that the most durable shales were those with low absorption values and high bulk specific gravities. Durability could also be evaluated by ethylene glycol immersion tests (Reidenouer, 1970). For assessing the durability of Oklahoma shales, a modified sand equivalent test was developed and was found to be very useful in distinguishing between soil-like and rock-like shales readily (Laguros, 1972).

Influence of Weathering on Shales

The tests indicated in the previous paragraph serve to identify the shales which are suitable for use in construction and not expected to create major problems during construction or during the service life of the pavement. These shales are defined as "no problem" shales and rock materials. However, their durability characteristics are slightly modified by weathering to varying degrees, depending on the factors influencing weathering. The factors, in the case of natural deposits, are the nature of the parent rock, climate, topography, vegetation and time (Grim, 1968). The weathering of materials used in construction is controlled by the nature of the material, compaction stresses, climate, drainage conditions, traffic stresses and time. All the factors except climate can be suitably chosen or accounted for in the design. However, with the added effects of temperature and rainfall, these factors undergo changes in a cyclic order, one being affected by another and slowly reduce the life of the pavements. Also, weathering has adverse effects on the engineering properties of the construction materials.

Chandler (1969) studied the effect of weathering on the shear strength properties of Keuper Marl. The changes in index properties and shear strength of Keuper Marl series found in different weathered conditions were investigated. Weathering zones were distinguished by visual examination of the particulate material.

In a similar study on Lisa clay (Chandler, 1972), an index of oxidation was formulated to indicate the degree of weathering. The index was defined as the ratio of Fe_2O_3/FeO, the relative proportions determined on a sample of 1 gm. The gradation became finer with the progress of weathering. The values of plasticity index increased and density decreased with weathering.

The experimental data showed a progressive change of the relationship between strength and water content with varying degrees of weathering. The natural water content increased with weathering and strength decreased.

Predictability of Weathering Changes

Knowing the effects of weathering on the "in-service" properties of the pavements, it is only desirable to predict the results of weathering and incorporate the consequent changes in the design processes. But it is recognized that it is impossible to reproduce the effects of natural weathering in the laboratory by physical means without considerable expense, time and some uncertainty. Moreover, the material breakdown in nature under varying types of exposure tends to be different. Still, it is hypothesized that the principles involved are the same.

Particles may be broken down more readily by the impact of heavy wheel loads than by the gentler impact of rain drops, but they still will break at their weakest points. The breakdown of the material is effected in multitude of

ways as described in various weathering and durability tests. The most common are wetting and drying, chemical attack, continuous leaching, abrasive action and impact of drop-hammer (Nettleton and Kiek, 1966). The ultrasonic treatment is a recent development for simulating the effects of weathering (Laguros, 1972).

Application of Ultrasonics

Ultrasonics has been successfully employed in so many different fields that the subject has developed into a major branch of engineering by itself. Only one aspect of such a vast area of specialization is of interest here and therefore background information is limited to this extent.

In general, high intensity applications of ultrasonics are those which produce changes in or effects on media, or the contents of the media, through which the waves propagate. The effects of the high intensity ultrasonic energy are (1) heat, (2) stirring, (3) cavitation, (4) chemical effects, and (5) mechanical effects, the most important one being cavitation (Ensminger, 1972).

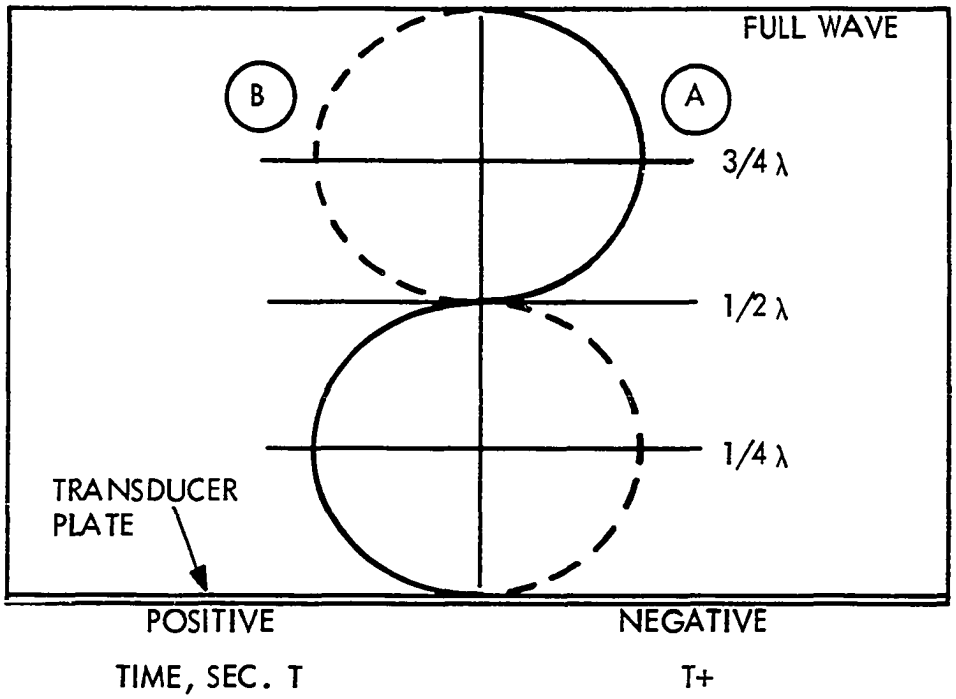
The high level of ultrasonic energy is produced by transducers which change the low frequency electrical energy into a high frequency (20,000 cps or higher) mechanical sound waves in either the probe type or the tank type equipment. In the probe type equipment, the probe transmits ultrasonic waves into the liquid medium directly. In the

tank type, the transducer is attached to the bottom of the tank and serves as a point of minimum pressure.

When the tank is filled with liquid to a depth, which is a multiple of a half wave length, mechanical sound waves are produced. These waves create alternatingly negative and positive pressures at any one point at intervals of half a cycle (Figure 2.1). As the negative pressure, i.e., pressure less than the vapor pressure of water, passes the point, it causes cavitation and half a cycle later, at the same point, a positive condition is created wherein the wave energy causes the vapor bubble to implode.

The quantity of energy released in any one implosion is extremely small; however, due to the corresponding small volumes involved, enormous pressures in the order of 10,000 psi and enormous temperatures in the order of 20,000°F are developed and dissipated (Westinghouse, 1968). Thus the cavitation, i.e., the formation and implosion of these vapor pockets resulting in high temperatures and pressures, constitutes the main effect of the disaggregation.

Stresses developed in an ultrasonic field can cause ruptures to occur in the materials or severe erosion of surfaces. They may also cause relative motion between surfaces thus producing selective absorption at these surfaces. When ultrasonic energy is applied to shales, cavitation causes the bonds between the individual particles making up the shale to break and disintegrate. Disaggregations of sandstones,



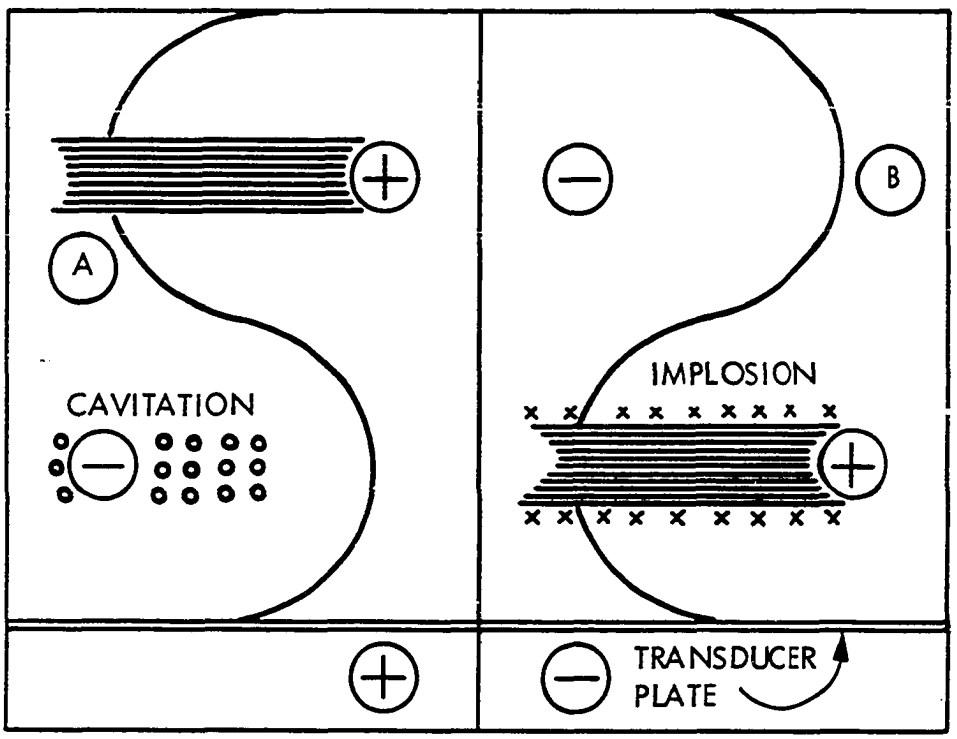


Figure 2.1 Standing pressure wave.

siltstones, and shales on application of ultrasonic energy were reported by Savage (1969), Gipson (1963) and Alguire (1969). Walker and McCarthy (1967) studied ultrasonic dispersion of clay bodies. Gipson (1963) used both the probe type and tank type ultrasonic units for the disaggregation of shales and found the tank type unit more suitable for compact shales and clay shales. Alguire (1969) used the tank type unit for ultrasonic disaggregation of some Oklahoma shales and found that the gradation and plasticity characteristics changed significantly for the ultrasonic treated material.

Engineering Properties of Shales

The literature on the engineering properties of shales was found to be limited. Since the shales contained large amounts of clays and silts, the literature pertaining to the engineering properties of compacted clays and silty clays becomes relative and pertinant and therefore it is presented herein.

Atterberg Limits

The liquid and plastic limit tests are very important tests in identification and classification of soils. The limit values characterize the soil-water relationships. The values are influenced by chemical, mineralogical and physical characteristics of soil constituents (Seed, Woodward and Lundgren, 1964). As the engineering behavior of soil depends

to a large extent on the amount, distribution and movement of moisture, the limit values are often correlated to other engineering properties.

Determined for shales, these limit values can also indicate the state of the shale or the degree of weathering between initial indurated condition and ultimate breakdown. The Atterberg limit values are also influenced by the method of sample preparation. Ultrasonic treatment increases the liquid limit and plasticity index values (Laguros, 1972). Natural weathering of shales also influence the limit values in a similar manner (Laguros, Kumar and Annamalai, 1974).

Moisture- Density and Void Ratio

The natural moisture contents and bulk densities of shales vary with the degree of weathering. Values of low moisture content (5 to 15 percent) and high dry density (110 to 160 pcf) indicate well indurated "favorable" shale; on the other hand, values of high nature moisture content (20 to 35 percent) and low density (70 to 100 pcf) are associated with highly weathered "unfavorable" shales (Underwood, 1967). Void ratio, which is inversely related to the dry density, increases with weathering.

Strength

The compressive strength of shales varies over a very wide range from less than 25 psi for weaker compaction shales to more than 15,000 psi for well cemented shales. It depends on the

amount of compaction, type and amount of cementing agents, particle orientation and moisture content. Weathering would be expected to decrease the strength of soils as it adversely affects the above variables. Some of the "soil-like" shales exhibit high sensitivity due to disturbance in handling. They also behave in a similar manner as compacted clays.

Effect of structure: It is interesting to follow the viewpoints of various investigators regarding the structure of compacted clays. Lambe (1958) proposed a mechanistic model for the structure of compacted clays, considering possible arrangements composed of single clay plates (Figure 2.2a and 2.2b). Tan (1958) visualized the structure differently (Figure 2.2c). Aylmore and Quirk (1960) and Olsen (1962) observed that both artificial and natural clay soils were made up of clay plates aggregated into peds, clusters or domains and termed the structure as "turbostratic" (Figure 2.2d). The larger groups of approximately oriented particles were called "domains." A good example of this is "stack" (Figure 2.2f) which is in fact a large particle formed of perfectly oriented plates. Yong (1971) suggested that the single plate theory would be relevant only to dilute colloidal suspensions and that natural clays required consideration of multiple plate unit.

A recent electron microscope study of a large number of natural clay soils from various parts of the world (Barden, 1972; Barden and Sides, 1971) has revealed almost no

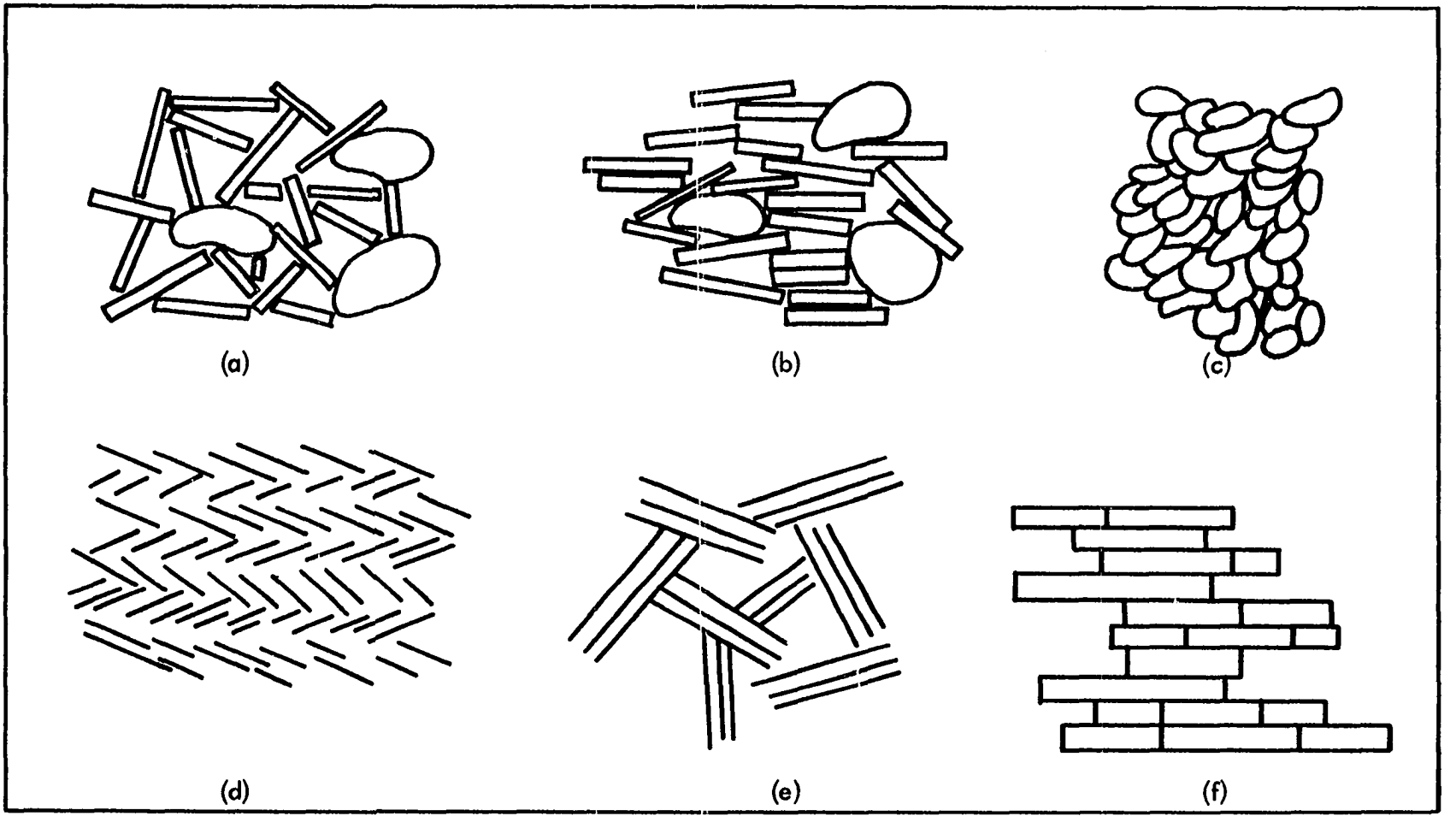


Figure 2.2 Idealized clay structures, (a) card house, and (b) dispersed, after Lambe (1958), (c) clay model, after Tan (1958), (d) turbostratic, after Aylmore and Quirk (1960) and (e) book house, and (f) stack, after Barden and Sides (1971).

single plate "cardhouse" structures but a general occurrence of plates aggregated face to face. In dispersed conditions this leads to "turbostratic" structures and in flocculated conditions to "bookhouse" structures (Figure 2.2e). Compacted clay has a macrostructure of large crumb-like aggregates and this granular macrostructure, rather than the microstructure, has been shown to govern most of the important engineering behavior of compacted clays.

Effect of compaction: The shear strength of compacted clays is influenced by the amount and nature of compactive effort, amount of water content and distribution of various size fractions of soil particles (Seed and Chan, 1959). The shear strains produced during compaction by different compaction methods are primarily responsible for the influence of the method of compaction on soil properties as it produces different structures. Barden and Sides (1970) noted a marked difference in structure, at low magnifications, between samples compacted dry of optimum and those compacted wet of optimum, but little difference had been observed at high magnifications. A flocculated structure results for water contents on the dry side of optimum and a dispersed structure on the wet side. Voids are continuous in both the flocculated and dispersed conditions. At optimum moisture contents, the air voids are found occluded.

In samples compacted wet of optimum, the method of compaction is influential. In samples compacted on the dry

side, the interparticle forces not being predominant, flocculation will occur under all compaction conditions but the degree of particle orientation will be dependent upon shear strains produced by compaction (Seed and Chan, 1959).

Effect of thixotropy: Elapsed time after compaction can cause significant changes in the properties of compacted clays. The thixotropic effects are pronounced over a range of water contents near the optimum moisture content. The time between sample preparation and testing should be controlled such that all tests are equally affected by thixotropy (Mitchell, 1964). After extensive research on artificially sedimented and leached clays, Gray and Kashmeeri (1971) concluded that the bulk of the thixotropic stiffening occurred within the first two weeks, with the rate of stiffening determined by the type of clay, the molding water content, and the electrolyte concentration.

Physicochemical effects: Ladd and Kinner (1967) investigated the influence of various physicochemical factors on the shear strength of clays. These factors are mainly the double layer osmotic repulsive force, van der Waal's attractive force and cementation of some natural clays due to carbonate, iron oxides and possibly organic matter. Shearing resistance to the applied energy is controlled by a mutual balance of the above factors. Mesri and Olsen (1970) studied the shear strength of montmorillonites and the results confirmed the above views. They noted that in

the case of pure clays, the shear strength is also affected by the nature of the adsorbed cations.

Effects of test conditions: The measured values of strength are also affected by the method of loading, rate of loading, strain level defining the strength, specimen size, drainage conditions, lateral pressure and base restraints (Lundgren, Mitchell and Wilson, 1968; Rowe and Barden, 1964; Duncan and Dunlop, 1968; Seed and Chan, 1959; Van Auken, 1963; and Perloff and Osterberg, 1963).

Lee and Shubeck (1968) compared the results of conventional triaxial tests and plane strain tests and concluded that the same failure strength was obtained for samples having the same final water content, irrespective of the test methods.

Theoretical considerations of strength mechanisms: Various mechanisms have been proposed for the development of shear strength by different investigators. The technique in general was that they started out with some hypothetical assumptions about the shape and size of the soil particles and water in the voids, drew conclusions from experimental data and proposed mechanism for the shear strength development.

Trollope and Chan (1960) observed a step-strain phenomenon in their data. During shear, the soil particles occupied stable and unstable positions alternately. Moving them from stable to unstable position required more energy, i.e., more stress, than moving them from unstable to stable

positions. Hence, the stress-strain curve consisted of a number of small jumps instead of being smooth.

Geuze (1964) assumed that the clay platelets made edge to face contacts with each other forming hinges and were oriented at a random fashion. With the progress of shear, the tensile bonds at edge to face contacts were broken continuously. The limit of structural strength is due to the rupture of all the tensile bonds. Beyond this limit, particle displacements take place in a continuous edge to face sliding of a viscous nature.

Murayama and Shibata (1964) also considered the contacts among the clay segments to be only edge to face, bound by thin layers of adsorbed water. They conceived two possible arrangements for clay segments, one forming an elastic joint and the other a viscoelastic joint. On the application of energy, the elastic joints collapsed to form viscoelastic joints. The shear strength, consolidation and rheological characteristics were considered in terms of these viscoelastic joints.

Considerable progress has been made in understanding the mechanism of clay deformation theory by the application of a rate process theory. The following investigators used this approach with different assumptions about the nature of interparticle bonds.

Noble and Demirel (1969) envisaged that (in a cohesive soil) interparticle bonds form due to an oriented

structure in the adsorbed water layer's adjacent clay particles. Deformation of the system, then, occurs by distortion and breaking of these bonds. The unstable condition that exists following the breaking of a bond is considered the "activated state" in which the "contact zone" between particles consists essentially of oriented water.

Mitchell, Singh and Campanella (1969) considered that interparticle bonds must be effectively solid to solid and since the individual atoms in the contact structure are the activating flow units, a contact may contain many interparticle bonds. They also suggested that the deformation of the systems occurred by distortion and breaking of these bonds.

Failure concepts: Failure in clay soils may occur either by flow or by localized material rupture. Generally, soils are considered to be frictional material and the Mohr-Coulomb theory is the most widely used theory in practice. The Mohr-Coulomb theory is based on the premise that the soil will fail or yield when either the obliquity of resultant stress reaches a maximum value on some plane in the material, or the maximum tensile normal stress reaches a value characteristic of the material.

The cohesive soil, in saturated condition, undergoes or sustains irrecoverable plastic deformation under very small applied loads and continues to deform throughout the loading process. Hence the theory of plasticity is thought

to be a good approach for the analysis of deformation problems involving clays. Drucker (1953) has interpreted both the Tresca (Maximum Shear Stress theory) and the von Mises (Distortion Energy theory or Octahedral Shear Stress theory) failure criteria (modified to be conical rather than cylindrical surfaces) in principal stress space. Both von Mises and Tresca failure conditions for axisymmetric triaxial tests become one and the same.

Yong and McKeyes (1971), after studying the yield and failure of clay under triaxial stresses, concluded that analytical plasticity techniques might be applied successfully to describe the undrained stress-strain behavior of a saturated clay up to a shear stress level of approximately half of the shear strength of the material. Beyond this, the deformation behavior deviated from that of a plastic material and approached that of a frictional medium, the ultimate stresses being describable in terms of Mohr-Coulomb failure theory.

Volume Change - Swelling Characteristics

Shale, when excavated and used subsequently in construction, experiences volume changes of varying degrees. The mechanisms involved in volume change or swelling are mainly physicochemical. They are controlled by the specific surface area, the type and concentration of ions, type and amount of clay and other cementing agents. The physicochemical properties of clays are modified by compaction and

molding water content as these control the particle spacing, degree of parallel orientation and capillary tension in the adsorbed layers (Bolt, 1956).

Seed, Woodward and Lundgren (1963), and Komornik and David (1969) have studied the swelling characteristics of compacted clays. Plasticity index, molding water content and density were correlated to either free swell or swelling pressures and consequently were proposed as indicative parameters for predicting the swelling behavior of the clays.

In another study on Oklahoma shales, Laguros (1972) has indicated that the amount of clay and clay sized particles of less than 2 micron size correlated well with the magnitude of volume change. The high coefficient of correlation (0.94) confirms that the volume change mechanism is chiefly physico-chemical and hence proportional to the clay content.

Consolidation Characteristics

Settlement problems associated with pavements and embankments formed with shale materials are not uncommon. These could be predicted well in advance from the results of consolidation tests. Considerable research has been done on the theory of consolidation and its application to clays existing in all states--unsaturated, saturated, undisturbed or remolded (Barden, 1965; Gibson, England and Hussey, 1967; and Schiffman, Chen and Jordan, 1969).

For undisturbed or remolded saturated clays, the one dimensional theory of Terzaghi is generally employed (Terzaghi

and Peck, 1967). The following simplifying assumptions are made:

1. The coefficient of permeability is the same at every point in the consolidating layer and for every stage of consolidation.
2. The coefficient of volume compressibility is the same at every point in the layer and for every stage of consolidation.
3. The excess water drains out only along vertical lines.
4. The secondary compression is neglected.

Consolidation of compacted and unsaturated clays has been investigated by Barden (1965). The porosity, the structure of clay and degree of saturation are identified to be the governing factors. He has observed that for the analysis of remolded unsaturated clays the Terzaghi theory of consolidation is sufficiently accurate if the degree of saturation is 90 percent or more. Bjerrum (1967) reported that the remolding and weathering changed the consolidation characteristics of overconsolidated clays and clay shales. The rebound curves were steeper for remolded clays than for undisturbed samples. This is due to the swelling as a result of broken bonds.

Sridharan and Venkatappa Rao (1973) found that the volume change behavior of saturated clays was controlled basically by two mechanisms which were governed by the modified effective stress concept. In mechanism 1, the volume

change was controlled by the shearing resistance at inter-particle level and in mechanism 2, primarily by the long range repulsive forces of the diffuse double layer. The experimental results from one dimensional consolidation tests on kaolinite and montmorillonite samples with different pore fluids indicated that mechanism 1 primarily governed the volume change behavior of non-expanding lattice type clays like kaolinite, and mechanism 2, that of the expanding lattice type clays like montmorillonite.

CHAPTER III

SELECTION AND PREPARATION OF SHALE SAMPLES

Selection of Materials

The selection of the shale samples for this investigation was based on the results of the study "Predictability of Physical Changes of Clay-Forming Materials in Oklahoma," (Laguros, 1972).

In the study, shale samples from 24 locations all over the state of Oklahoma were investigated. Based on the physical and physicochemical properties, the shales were classified into five distinct groups. Six shale samples from these five groups (two from one group and one from each of the other four groups) were selected for intensive study. Durability tests and ultrasonic tests were performed extensively on these six shale samples. Based on their response to ultrasonic treatment and type of predominant clay mineral, four of the six shale samples were selected for this investigation. The sampling locations of these shales are indicated in Figure 3.1. The physical and physicochemical properties of these four shales are presented in Table 3.1. They belong to different geologic systems.

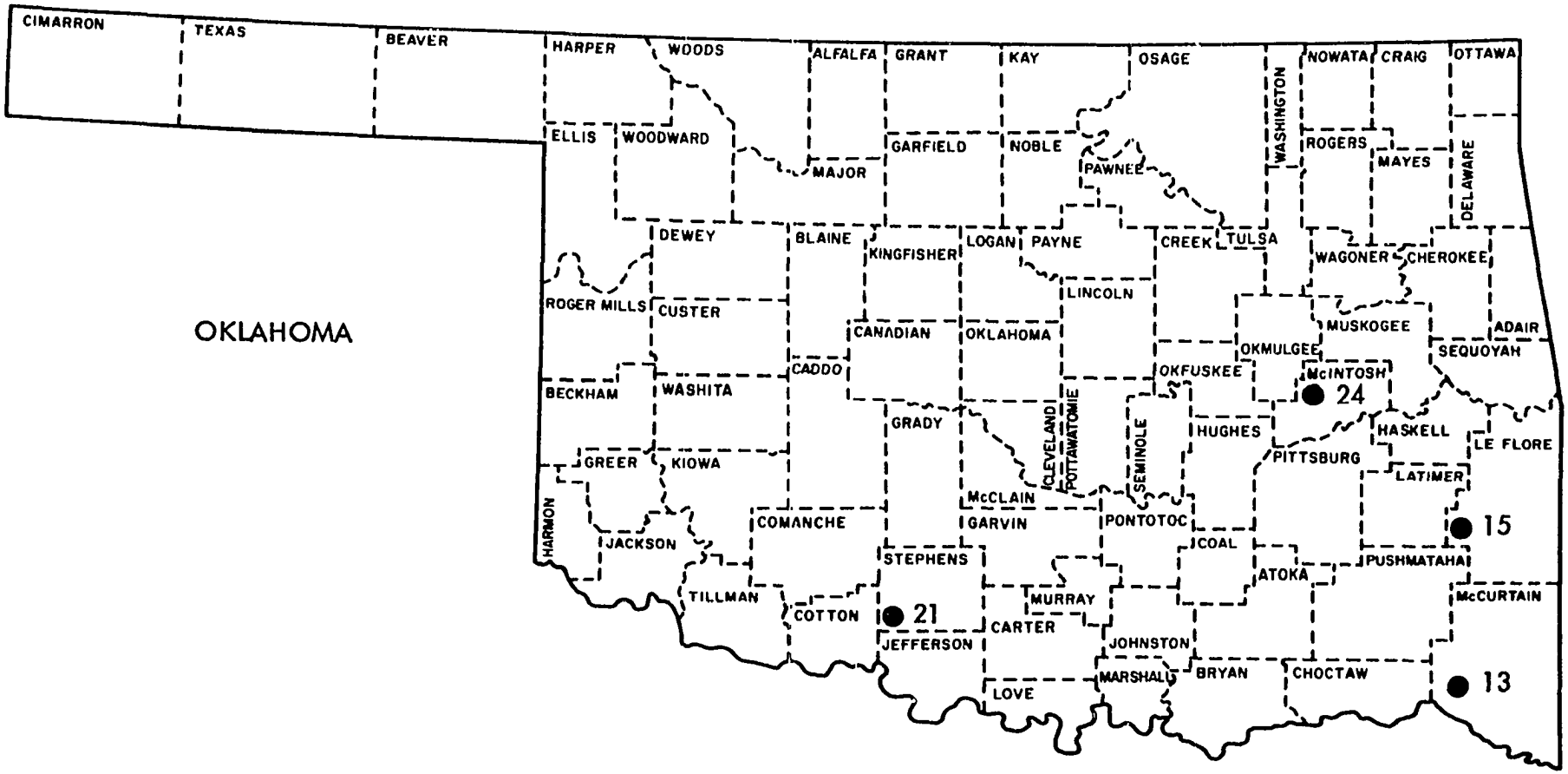


Figure 3.1 Location and identification of selected shale samples.

TABLE 3.1

PROPERTIES OF SHALES

Shale No.	Geologic Particulars			Textural Composition[b] (%)				Physical			Chemical			Classification		
	Location (County)	Geologic System[a]	Oklahoma Geologic Unit	Sand (2.0-0.074 mm)	Silt (0.074 -0.005 mm)	Clay (0.005 mm)	Clay (0.002 mm)	LL[c] (%)	PI[d] (%)	S.G[e] ($\frac{gm}{cc}$)	CEC[f] $\frac{meq}{100gm}$	pH[g]	Predominant Clay Mineral[h]	Textural[i]	Unified[j]	AASHO[k]
13	McCurtain	Cretaceous	Washita	5	32	63	52	47	26	2.73	20	5.1	M*	Clay	CL	A-7(6)
15	LeFlore	Mississippian	Stanley	69	18	13	9	33	13	2.77	20	7.9	I*	Sandy Loam	ML	A-1-b
21	Stephens	Permian	Claypool	3	53	44	28	29	8	2.79	21	8.5	ML*	Clay	ML	A-6
24	McIntosh	Pennsylvanian	Senora	44	21	35	23	38	13	2.73	22	7.6	I	Clay	ML	A-4

[a] From Reference (Sheerar, 1932).

[b] AASHO Method T 88-57.

[c] AASHO Method T 89-60.

[d] AASHO Method T 90-61.

[e] AASHO Method T 100-60.

[f] Cation exchange capacity determined by continuous titration method (Kerns, 1967).

[g] Glass electrode method using 10 gm of shale in 50 cc distilled water.

[h] X-ray diffraction analysis.

[i] Triangular chart, U.S. Bureau of Public Roads.

[j] TM No. 3-357 Method, Corps of Engineers.

[k] AASHO Method M 145-49.

* M - Montmorillonite; I - Illite; ML - mixed montmorillonite-illite.

According to the AASHO classification system, each is different. They are also those shales, found in abundance in the state and are used extensively in highway construction.

Preparation of Raw or Untreated Shale Samples

All the shale samples, brought from the field, were air dried and crushed with pestle and mortar to pass through U.S.Std. 3/8 in sieve. The material passing the sieve is fed into a soil grinder and crushed to pass through U.S.Std. Sieve number 10. All the tests on raw or untreated shale samples were done on this material.

Preparation of Ultrasonic Treated Shale Samples

Apparatus

A tank type of ultrasonic equipment was used as this type is the most suitable for ultrasonic disaggregation of clay shales and clays. A Westinghouse Mini Magnapak ultrasonic cleanser was used. The tank was 9-1/2 in long, 6 in wide and 6 in deep with a capacity of 1.5 gal. The generator had an average output power of 200 w (3.5 w/sq in) and frequency of 20 kc. A complete set-up of the ultrasonic equipment is shown in Figure 3.2.

Preliminary Investigations

Many pilot studies were carried out to standardize the ultrasonic treatment as applied to shales in the laboratory. It is of interest to note that this type of equipment is used

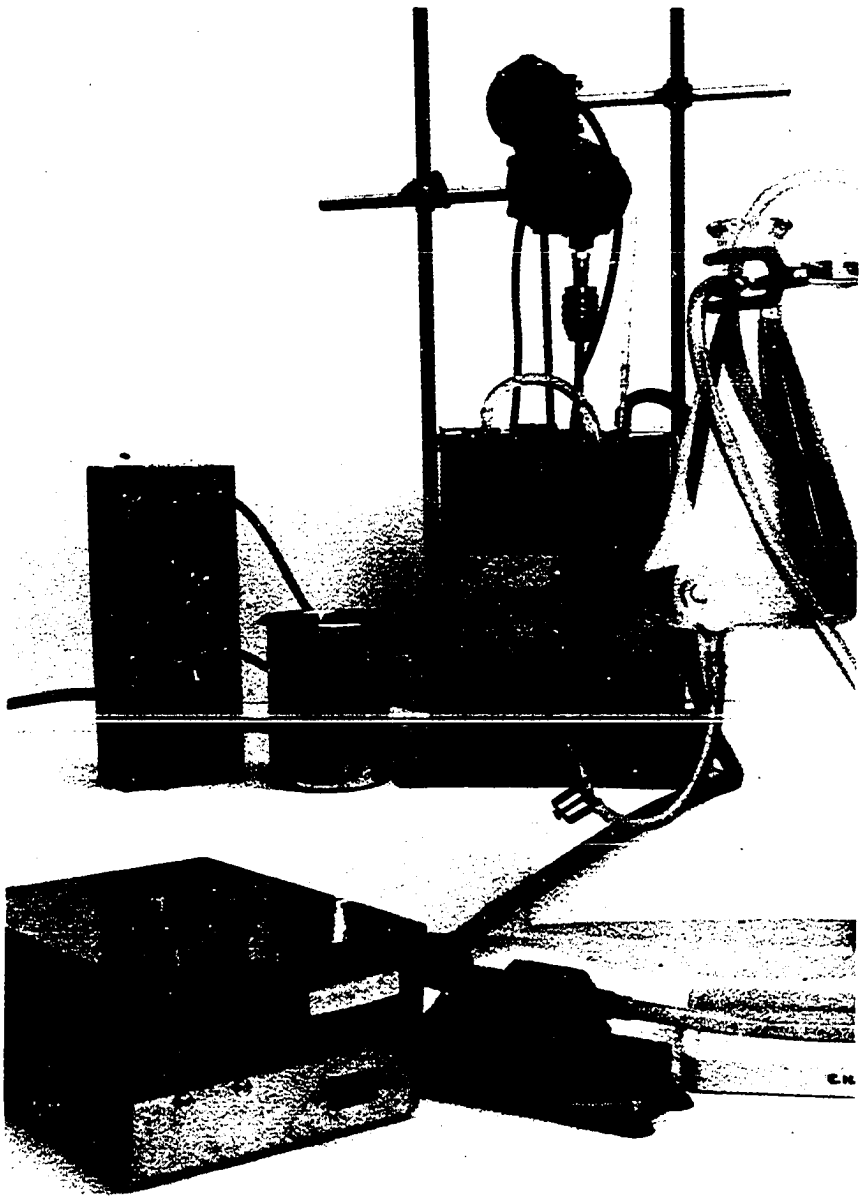


Figure 3.2 General view of ultrasonic equipment and water circulation system.

to remove soil impurities in metallic parts. The cleaning could be done within a period of one to four minutes, depending on the power input and the ratio of volume of material to be cleaned to the volume of liquid medium in the tank.

However, shales could not be kept directly inside the ultrasonic tank as it was difficult to transfer the treated shale samples. Hence, the shales were first taken in separate stainless steel beakers. The beakers were then kept inside the ultrasonic tank. The stainless steel beakers made good metal-metal contacts with the bottom of the ultrasonic tank. They served as independent ultrasonic tanks, as cavitation was found to take place inside the beakers. But the rate of formation of cavitation was rather slow and ultrasonic energy, transmitted to the shale was less. Increasing the time of ultrasonic treatment and reducing the sample size offset this limitation.

Also, during ultrasonic treatment, the temperature of water inside the tank reached high levels, i.e., about 180°F, affecting the clay minerals. To avoid this excessive temperature, it was controlled at 70° ± 2°F by circulating water around the beakers in the ultrasonic tank. It may be doubted if circulation of water would interfere with the cavitation. The circulation was found not to affect the process of cavitation inside the beakers as these themselves acted as mini ultrasonic tanks.

It was found that most of the shale samples adhered to the bottom of the beakers after treatment and transference took much time and large quantities of wash water. Mechanical stirring at a very low speed (4 to 6 revolutions per minute) was found to dislodge the shale material and keep all the material in suspension during treatment. It is possible that the mechanical stirring might have some effect on the cavitation process inside the beakers and also cause additional breakdown of the material.

However, the stirring is inevitable for practical purposes. At very low rate of stirring such as 4 to 6 revolutions per minute, cavitation was found to take place inside the beakers and did not seem to be affected by stirring. The combined effect of both ultrasonic treatment and mechanical stirring is considered as the effect of ultrasonic treatment.

Treatment Time

In the study (Laguros, 1972), the shale samples were subjected to ultrasonic treatment for time durations of 1/4, 1/2, 1, 2, 4 and 8 hours and variations in the index properties with ultrasonic treatment time were studied. The changes in particle size distribution and Atterberg limits occurred rapidly within the first hour and then reached asymptotically stable values with time (Laguros, 1972). (See Figure 3.3).

As a part of this study, these shale samples were exposed to natural weathering for a period of 24 months.

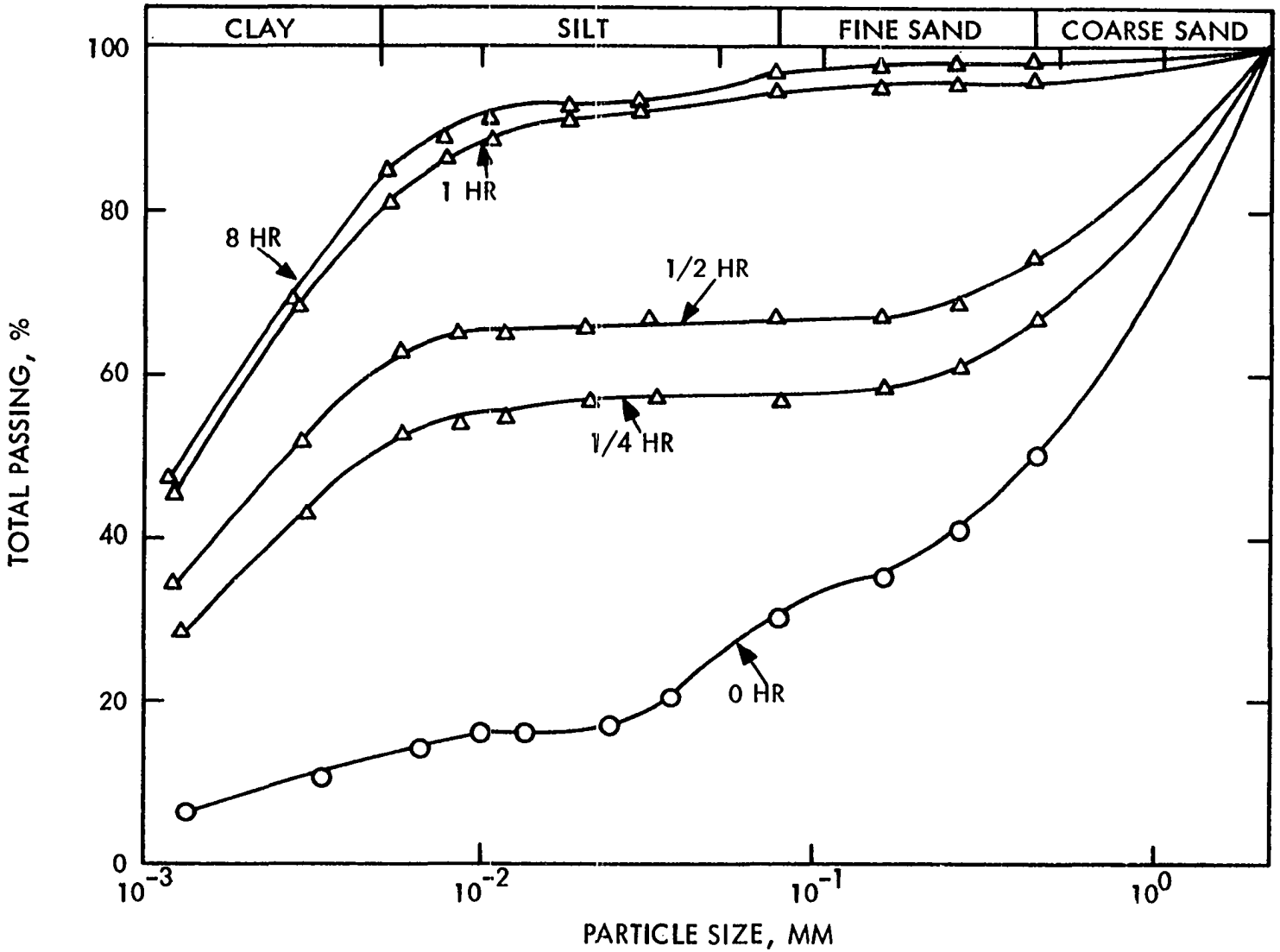


Figure 3.3 Effect of ultrasonic treatment time on gradation characteristics for shale 15, after Laguros (1972)..

The changes in the index properties were studied and compared with those obtained for different ultrasonic treatment times. As indicated in Table 3.2, the changes produced by 24-month field weathering in the index properties were found, generally, to correspond to those brought about by one hour or less of ultrasonic treatment (Laguros, Kumar and Annamalai, 1974).

These studies indicated that the optimum time of ultrasonic treatment was one hour. Hence, it was decided to use the same time in this investigation for ultrasonic treatment of the shale samples.

Ultrasonic Treatment Procedure

All the shale samples were air dried and prepared as for raw shale samples. The material passing U.S. Std. sieve number 10 was used.

A sample of 125 gm was soaked in 125 ml of distilled water for 12 hours or more. The sample was transferred to the stainless steel beaker using a minimum quantity of water. A duplicate sample was prepared likewise. The two beakers were kept inside the tank and water circulated around the beakers. The stirrers were introduced into the beakers and adjusted to stir at a very low rate of 4 to 6 revolutions per minute. The ultrasonic generator was turned on and the ultrasonic energy applied for one hour.

After treatment, the stirring and circulation were stopped, the beakers removed and the material was transferred into a glass dish. The soil water suspension was dried in

TABLE 3.2

INDEX PROPERTIES OF RAW, ULTRASONIC TREATED AND FIELD WEATHERED SHALE SAMPLES

Shale No.	8	13	15	21	22	24
5-micron clay (%)						
A	3	59	18	39	82	23
B	18	68	80	76	92	79
C	33	69	83	77	90	85
D	9	61	20	55	83	44
2-micron clay (%)						
A	2	48	14	25	63	14
B	11	57	60	54	78	57
C	23	58	61	62	77	65
D	6	53	15	48	63	29
Liquid Limit						
A	np	43	24	40	64	29
B	32	51	45	33	69	47
C	40	55	46	45	72	47
D	22	51	28	43	64	32
Plasticity Index						
A	np	23	2	14	29	6
B	np	29	20	13	34	20
C	16	32	20	23	39	22
D	np	33	7	20	32	7

A = raw sample; B = sample treated for 1 hour by ultrasonic method; C = sample treated for 8 hours by ultrasonic method; D = sample afforded 24-month natural weathering.

np = non plastic.

an oven at 140° ± 2°F. Excessive temperature was avoided. When dry, the soil was crushed to pass through U.S. Std. 3/8 in sieve and ground in the Hewitt soil grinder to pass U.S. Std. sieve number 10. For all shales, this procedure was followed to obtain ultrasonic treated shale samples and used in all the engineering tests except for gradation analysis and Atterberg limits.

For gradation analysis a 50 gm portion of the material passing U.S. Std. sieve number 10 was soaked in 125 ml of calgon solution for 12 to 18 hours. Then it was transferred to the stainless steel beakers and ultrasonic treated for one hour duration. After treatment, the soil solution was transferred to the hydrometer jar and the gradation test was performed.

Samples for Atterberg limit tests were prepared by soaking 125 gm of the shale material passing U.S.Std. sieve number 10 in 125 ml of distilled water for 12 to 18 hours and treating them ultrasonically for one hour. Then the material was washed through U.S.Std. sieve number 40, oven dried at 140° ± 2°F and pulverized again the pass U.S.Std. sieve number 40. This material was used for the determination of liquid and plastic limits.

CHAPTER IV

EXPERIMENTAL INVESTIGATIONS

The tests selected for evaluating the effectiveness of ultrasonic treatment were only those which are generally used for the identification and selection of highway soil materials and for design purposes. They are described in this chapter. All the tests were done on both raw and ultrasonic treated shale samples as per standard specifications mentioned thereof.

Grain Size Analysis

Grain size distribution was determined in accordance with the ASTM Designation D 422-63 (AASHO Designation T88-57). Calgon was used as the dispersing agent. Iowa jet dispersion apparatus was used to disperse the soil particle under an air pressure of 10 psi for about five minutes.

Specific Gravity

The specific gravity of the shale samples were determined in accordance with the ASTM Designation D854-65 (AASHO Designation T100-60).

Atterberg Limits

The liquid limit tests were run in accordance with the ASTM Designation D423-66 (AASHO Designation T89-60). The plastic limit tests were run in accordance with the ASTM Designation D424-65 (AASHO Designation T90-61).

Moisture-Density Tests

These tests were run in accordance with the ASTM Designation D698-66 (AASHO Designation T99-61). The only deviation from the standards was the compaction apparatus that was used. The Harvard Miniature Compaction apparatus was used instead of Proctor mold. The samples were compacted in five layers under a compactive effort of 25 blows per layer, using a 20 lb spring loaded rammer. The main advantage in using the Harvard method is that it requires only 1.5 lb of soil sample in contrast to about 15 lb required for standard Proctor test. Also, this method has to be used since the sample size is limited by time involved in the preparation of ultrasonic treated samples.

Triaxial Compressive Strength

The strength parameters are generally determined by direct shear, simple shear or triaxial compression tests. The advantage of triaxial compression tests is that the field conditions prior to and during construction can be duplicated in the laboratory to study the behavior of the soil. The general Mohr-Coulomb failure law is expressed by the formula

$$\tau = c + \sigma \tan \phi \qquad (4.1)$$

where τ = shear stress

c = cohesion

σ = normal total stress

ϕ = angle of friction

The normal total stress σ includes a number of parameters or terms and for a generalized soil-air-water system it may be expressed as

$$\sigma = \bar{\bar{\sigma}} \cdot a_m + u_a \cdot a_a + u_w \cdot a_w + A - R \qquad (4.2)$$

where $\bar{\bar{\sigma}}$ = contact stress at the contact points of mineral-mineral

a_m = (area of mineral-mineral contact)/(total area)

u_a = pore air pressure

a_a = (area of air-mineral contact)/(total area)

u_w = pore water pressure

a_w = (area of water-mineral contact)/(total area)

A = net attractive forces existing between clay platelets

R = net repulsive forces between clay platelets

However, A and R cannot be isolated and measured experimentally but considered to be predominant in dispersed plastic clays. For other textured soils A and R are usually ignored. Also, it is assumed that $\bar{\bar{\sigma}}$ is very large, $a_m \approx 0$ but $\bar{\bar{\sigma}} \cdot a_m$ is finite and equal to $\bar{\sigma}$ (effective stress), and $a_a + a_w \approx 1$.

For partially saturated soils, Equation 4.2 is written as

$$\sigma = \bar{\sigma} + u_a + a_w(u_w - u_a) \tag{4.3}$$

and for saturated soils,

$$\sigma = \bar{\sigma} + u_w \tag{4.4}$$

In terms of the effective stress, the Mohr-Coulomb equation is given as

$$\tau = \bar{c} + \bar{\sigma} \tan \bar{\phi} \tag{4.5}$$

where $\bar{c}$ = true cohesion

$\bar{\sigma}$ = effective normal stress

$\bar{\phi}$ = true angle of friction

Both c,ϕ and $\bar{c},\bar{\phi}$ are frequently employed in the design, depending on the actual field conditions.

In the present investigation, all triaxial compression tests were run as unconsolidated-undrained tests without pore water pressure measurement for the following reasons.

1. In the construction of an embankment or excavation of a slope, the stability of slopes is often governed by undrained shear strength of the soil as the soil is stressed quickly and no time is allowed for a drained condition to be established and the dissipation of pore water pressure.

2. In bases and subbases, the thickness involved being small, the changes in pore water pressures are not critical in strength determinations. Strength parameters determined from undrained tests are used for design of pavements.

For each shale sample, eight different series of tests were run varying the molding moisture content and rate of deformation. Descriptions of the variables used in each series are given in Table 4.1

Preparation of the Mix

The quantity of shale sample enough for any one series is mixed with calculated amount of distilled water, depending on the molding moisture content desired. The calculated amount of water also includes about 1 percent more water to allow for evaporation losses during mixing and handling. The mix is stored in plastic bags sealed with cellotape and moisture cured at near 100 percent relative humidity in glass desiccators with water in the bottom for a minimum period of two weeks.

Preparation of Test Specimens

After curing, the mix is thoroughly worked to destroy any thixotropic effect and then compacted into specimens using the Harvard Miniature Compaction apparatus. It was found very difficult to get good specimens from shale 13 at higher moisture contents and also from ultrasonic treated samples. For these series alone, samples were made using a

TABLE 4.1

VARIABLES USED IN THE TRIAXIAL TESTS AND TEST DESIGNATION

Designation of Test Series	Shale Sample Used	Strain Rate in/min	Molding Water Content
R-1	Raw	0.025	3% less than OMC* determined for raw shale.
R-2	Raw	0.025	OMC determined for raw shale.
R-3	Raw	0.025	3% more than OMC determined for raw shale.
U-1	Ultrasonic	0.025	OMC determined for raw shale.
U-2	Ultrasonic	0.025	OMC determined for ultrasonic treated shale.
R-2(S)	Raw	0.0025	OMC determined for raw shale.
U-1(S)	Ultrasonic	0.0025	OMC determined for raw shale.
U-2(S)	Ultrasonic	0.0025	OMC determined for ultrasonic treated shale.

*OMC - optimum moisture content.

static compaction apparatus. The apparatus is designed to have the same height to diameter ratio and produce the same density as the specimens from Harvard Miniature Compaction apparatus. The dimensions of the specimen are 1.352 in diameter and 2.910 in height as compared to 1.280 in diameter and 2.720 in height for the specimens compacted in the Harvard Miniature Compaction apparatus. The dimensions are not so significantly different as to cause size effect on the strength.

The specimens were wrapped with Saran Wrap and moisture cured for 24 hours prior to testing to bring about even distribution of moisture throughout the specimens. After equilibriation, the samples were measured for their dimensions and weights and recorded.

Testing

The specimens were tested in Clockhouse triaxial testing machine. The set-up is shown in Figure 4.1. The loading capacity of the machine is 10,000 lb and is capable of constant deformation of 0.00007 in/min to 0.16 in/min. The machine is modified to apply loads pneumatically also for constant rate of stress tests. The liquid used in the pressure cell is a 75 percent - 25 percent mixture of distilled water and glycerine, respectively. The constant for the proving ring used is 4.68 lb/division. Different cell pressures of 0, 10, 20 and 30 psi were used.

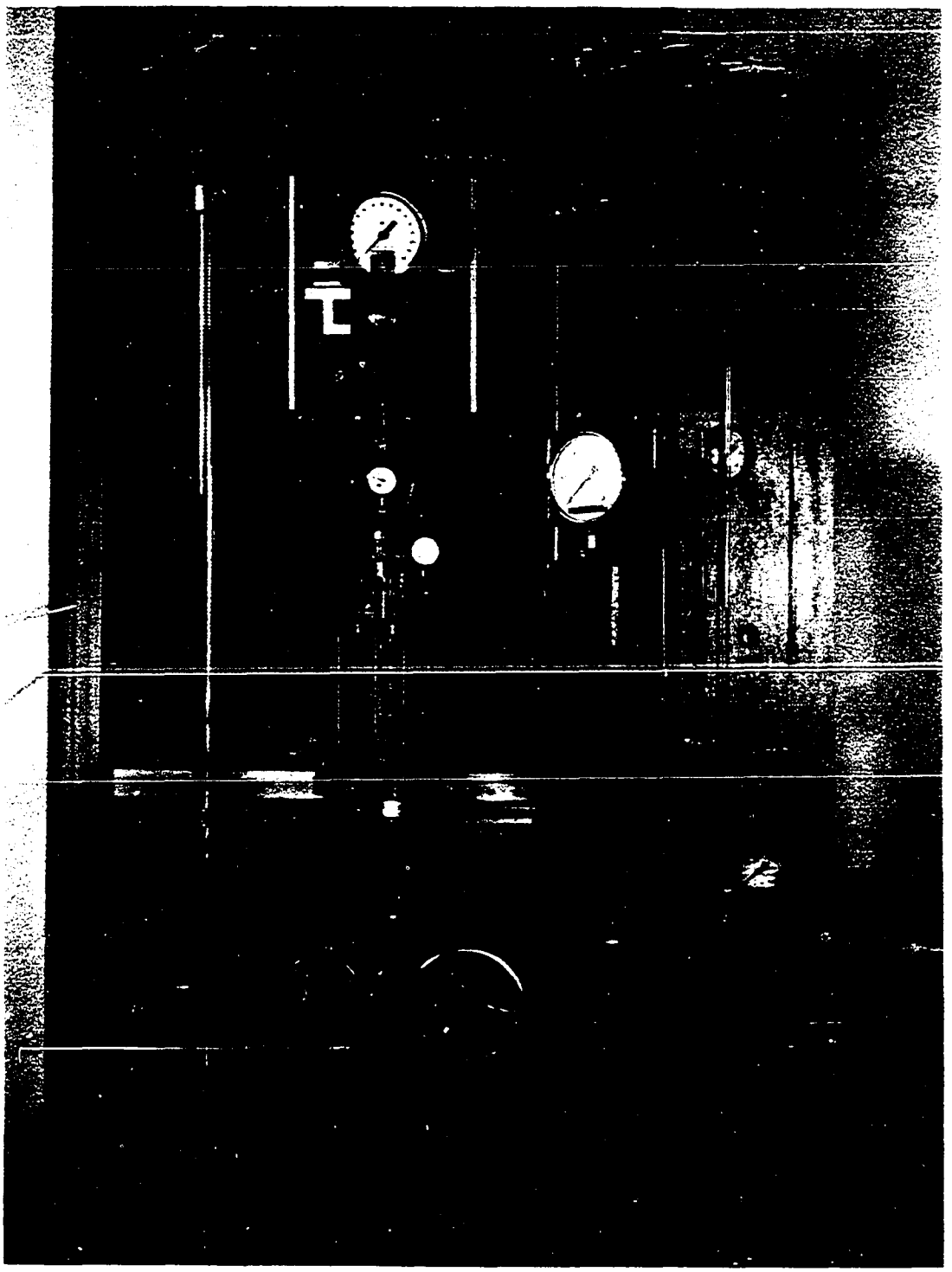


Figure 4.1 Triaxial compression test set up.

Relaxation Tests

Preparation of Specimen

The specimens were prepared and cured exactly in the same manner as for triaxial tests, the molding water content being the optimum moisture content as determined in the moisture-density tests.

Testing

Determination of strain level: It was decided to load the specimens up to one half on the maximum strain level and then allow to relax. The values of the strain level were obtained as follows. From the results of triaxial shear tests at cell pressures of 10, 20 and 30 psi for both raw and ultrasonic treated shale samples, the values of maximum strain level and hence the values of one half of the maximum strain level were calculated. The average value was found separately for raw shale and ultrasonic treated shale. Then, the strain level for the relaxation tests was chosen to be the minimum of the two average values.

Test conditions: The cell pressures used were 10, 20 and 30 psi. Loading was discontinued after reaching the one half maximum strain level. The readings of both load dial and deformation dial were taken at 0, 1/4, 1/2, 1, 2, 5, 10, 15 and 30 minutes of elapsed time and continued at 30 minute intervals until the load dial reading remained the same over a period of 30 minutes. Two rates of speed, 0.025 in/min and 0.0025 in/min, were used to load the specimens up to the chosen strain level.

Consolidation Test

Sample Preparation

The material used in the preparation of specimens for consolidation tests was prepared in a similar way as for strength tests. The amount of water used corresponded to the optimum moisture content determined by the moisture-density relationships.

A portion of 450 gm of the prepared shale sample was hand mixed with an amount of distilled water to bring the mixture to near optimum moisture. The mix was compacted in a Proctor mold with a compactive effort of 25 blows from a 5.5 lb rammer being dropped through 12 in. This conforms to AASHO standard method T99-61 for specimens prepared with single placement of soil in the mold.

The specimen was removed from the mold by means of a hydraulically operated extruder and hand trimmed using a trimming apparatus to fit 2.5 in diameter and 1.0 in high brass floating ring. The excess soil was trimmed to make its exposed surfaces flush with the face of the ring. The specimen along with brass floating ring was sandwiched between two 2.5 in diameter and 0.5 in thick porous stones and the whole assembly was immersed in distilled water for 24 hours to assure saturation.

At the end of immersion, the assembly was taken out, porous stones removed; any excess soil due to volume change was also removed. All the necessary weights were recorded

before and after immersion to facilitate computations of density, void ratio and moisture content.

Apparatus

The tests were performed in a levermatic consolidation apparatus using 2.5 in diameter and 1.0 in high floating ring, shown in Figure 4.2 (Soil Test model No. C-220). It included a dial indicator to record vertical deformations on loading and a weight set to give equivalent loading pressures from 1/16 tsf to 8 tsf with a load increment ratio of one.

Testing

The saturation of the sample during testing was obtained by flooding the specimen and porous stones at suitable intervals as suggested by Anessi (1970) in order to minimize the loss of soil between the bottom porous stone and the floating ring. Also, the consolidation period for the load increments was selected, based on the deformation rate, so as to cover only the range of primary consolidation as the study of the effects of secondary consolidation was outside the scope of the present investigation.

A complete consolidation test consisted of a loading cycle of 0 to 1/4, 1/4 to 1/2, 1/2 to 1, 1 to 2, 2 to 4 and 4 to 8 tsf load increments and an unloading cycle of 8 to 4, 4 to 2 and 2 to 1/8 tsf load decrements.

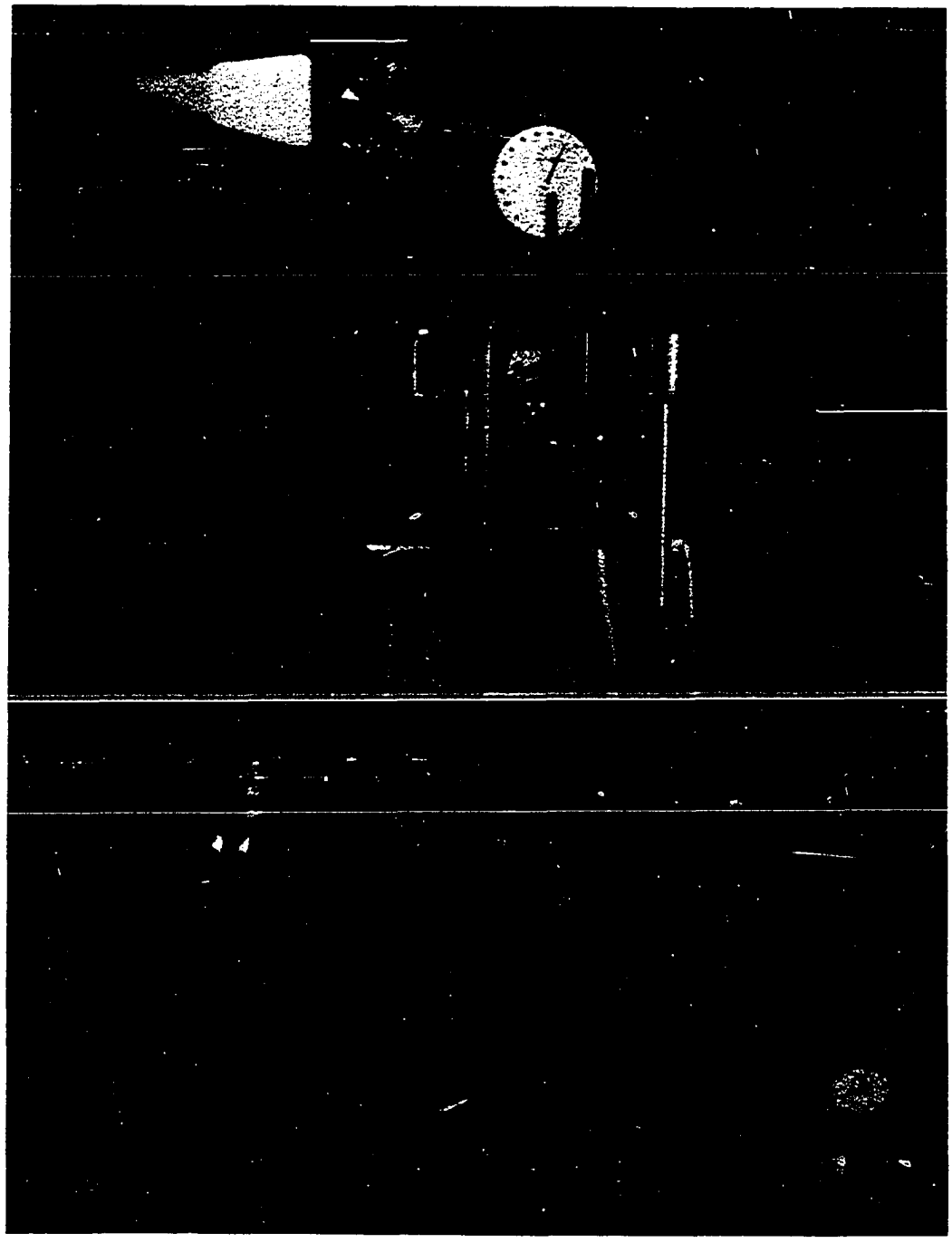


Figure 4.2 Levermatic consolidation testing device.

The period of test under each increment or decrement of load was 4 hours except for loading ranges of 1 to 2, 2 to 4 and 4 to 8 tsf. Under these increments, the tests were continued up to 12 hours as consolidation under high loading pressures took much longer time. Complete records of both loading and unloading data were maintained.

Volume Change Tests

The volume change tests were done according to ASSHO standards T-116-54.

Sample Preparation

The material passing U.S. Std. sieve number 10 was used for both raw and ultrasonic treated shale samples. Based on mix calculations for maximum dry density, sufficient amounts of dry soil and distilled water were mixed for obtaining a trimmed specimen 4 in diameter and 1.5625 in height. The mix was placed in the mold and compacted by 25 blows of a 5.5 lb rammer dropped from a height of 12 in above the soil. Then the specimen was trimmed flush with the edge of the mold.

Apparatus

The volume change apparatus consisted of a bottom plate, a mold, collar and a brass ring with a guided piston. The bottom plate and the brass ring were provided with suitable recesses to accommodate porous stones which, when assembled, were on either side of the compacted soil sample.

A tripod resting on top of the upper mold supported a dial indicator. The set-up is shown in Figure 4.3.

Test Procedure

After compaction and trimming, the brass ring with the porous stone was placed over the compacted sample. The tripod was positioned and the dial gage adjusted. Distilled water enough to maintain a water level of 1.0 in above the specimen was added into the upper mold. The volume change of the specimen on saturation was transmitted through the vertical movements of the piston and was measured by the dial indicator. Dial gage readings were taken for every 15 minutes for the first hour from the instant water was added into the upper mold. Then the readings were continued to be recorded hourly for the first 24 hours and afterwards for every 12 hours until the movement of the dial was equal to or less than 0.001 in over a period of 18 hours. Moisture content determinations before and after the test were made.

X-Ray Diffraction Studies

Sedimented slides were prepared for raw shale and ultrasonic treated shales as described herein. A portion of the material passing U.S. Std. sieve number 200 was mixed with 100 ml of distilled water in a measuring jar, thoroughly shaken and placed undisturbed. After 15 min of settling, the soil-water solution, containing 10 to 20 micron clay and clay sized particles, was pipetted out on

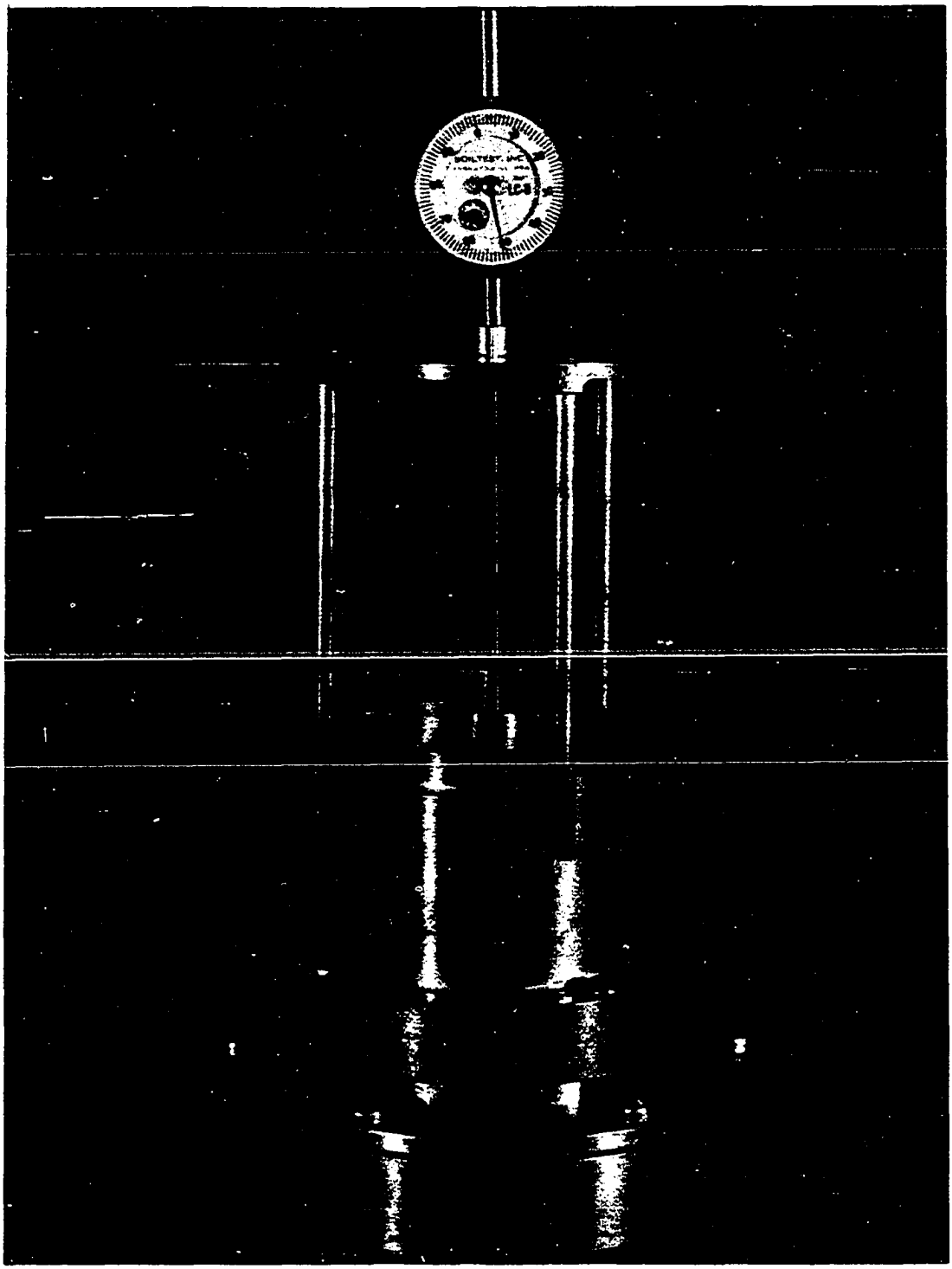


Figure 4.3 Volume change test apparatus.

the glass slide and allowed to dry in an oven at 140 ± 2°F.

X-ray diffraction patterns of these sedimented slides were obtained using either a Norelco x-ray diffractometer operated at 40 kv and 20 mA or a Siemens x-ray diffractometer operated at 35 kv and 18 mA. In both units, Cu-K_α radiation was used. All the patterns were run for diffraction angle of 2θ ranging from 3 degrees to 60 degrees. Glycolation and heating up to 600°C were used to confirm the clay minerals identified. The patterns for glycolated and heated slides were run only from 3 degrees to 13 degrees to check the first order peaks of typical clay minerals.

CHAPTER V

PRESENTATION AND DISCUSSION OF TEST RESULTS

In this chapter, the results of gradation, specific gravity, Atterberg limits, moisture-density, triaxial compression, relaxation, volume change, consolidation and x-ray diffraction tests on four shale samples before and after ultrasonic treatment are presented. The effect of ultrasonic treatment on the soil properties measured by the above tests is discussed.

Additionally, it became desirable to establish relationships among some of the engineering properties of shales, in that the scope of the treatment is to provide a predictive tool in the behavior of shale after ultrasonic treatment. Therefore, most of the data on the 24 shales which exhibit different degrees of weathering were obtained from a previous study (Laguros, 1972) and employed as a means of a broader basis for correlation analysis.

Grain Size Analysis

The grain size distribution curves for raw and ultrasonic treated shales are presented in Figures 5.1 through 5.4. The amounts of silt, less than 5 micron and 2 micron

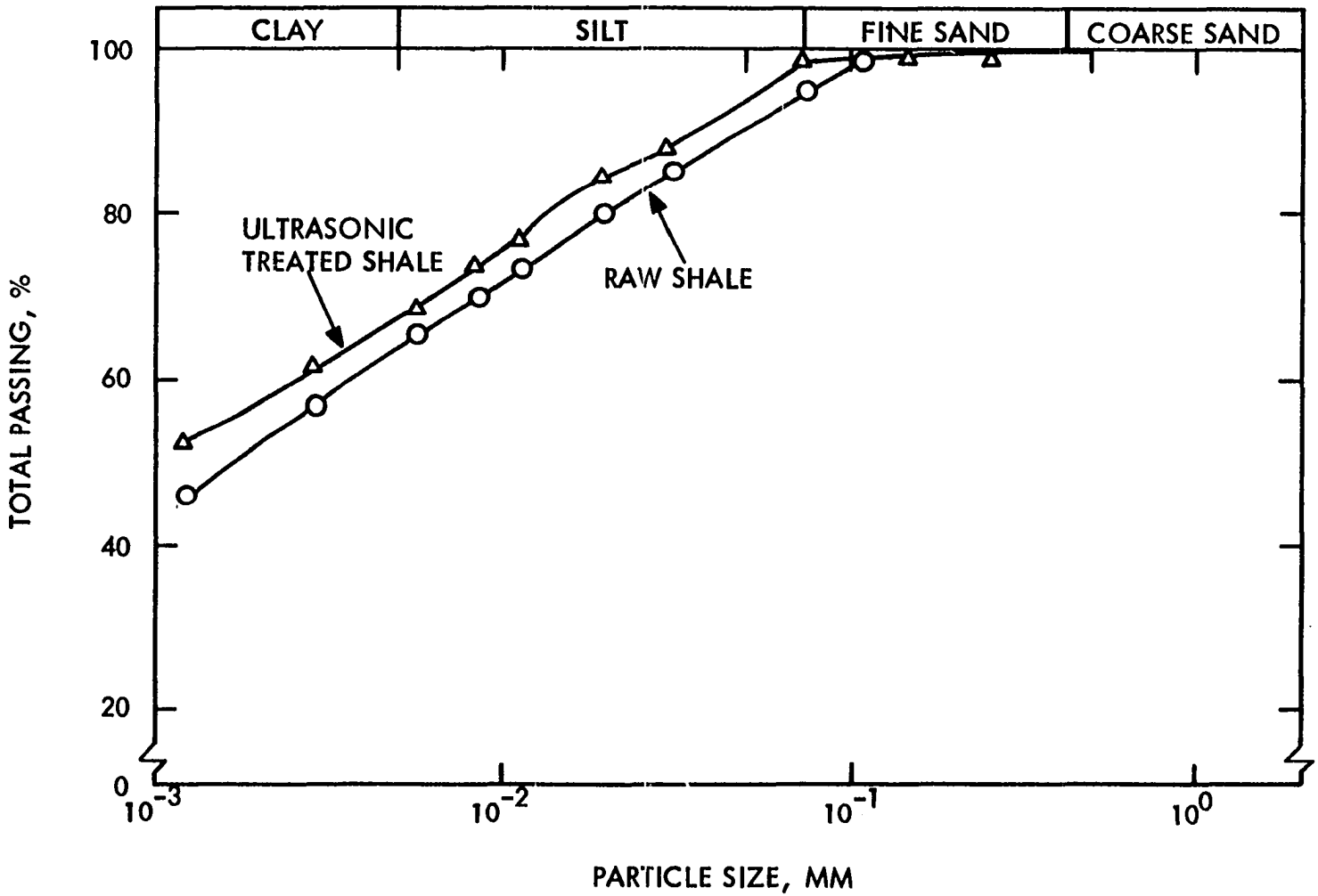


Figure 5.1 Comparison of gradation characteristics for shale 13.

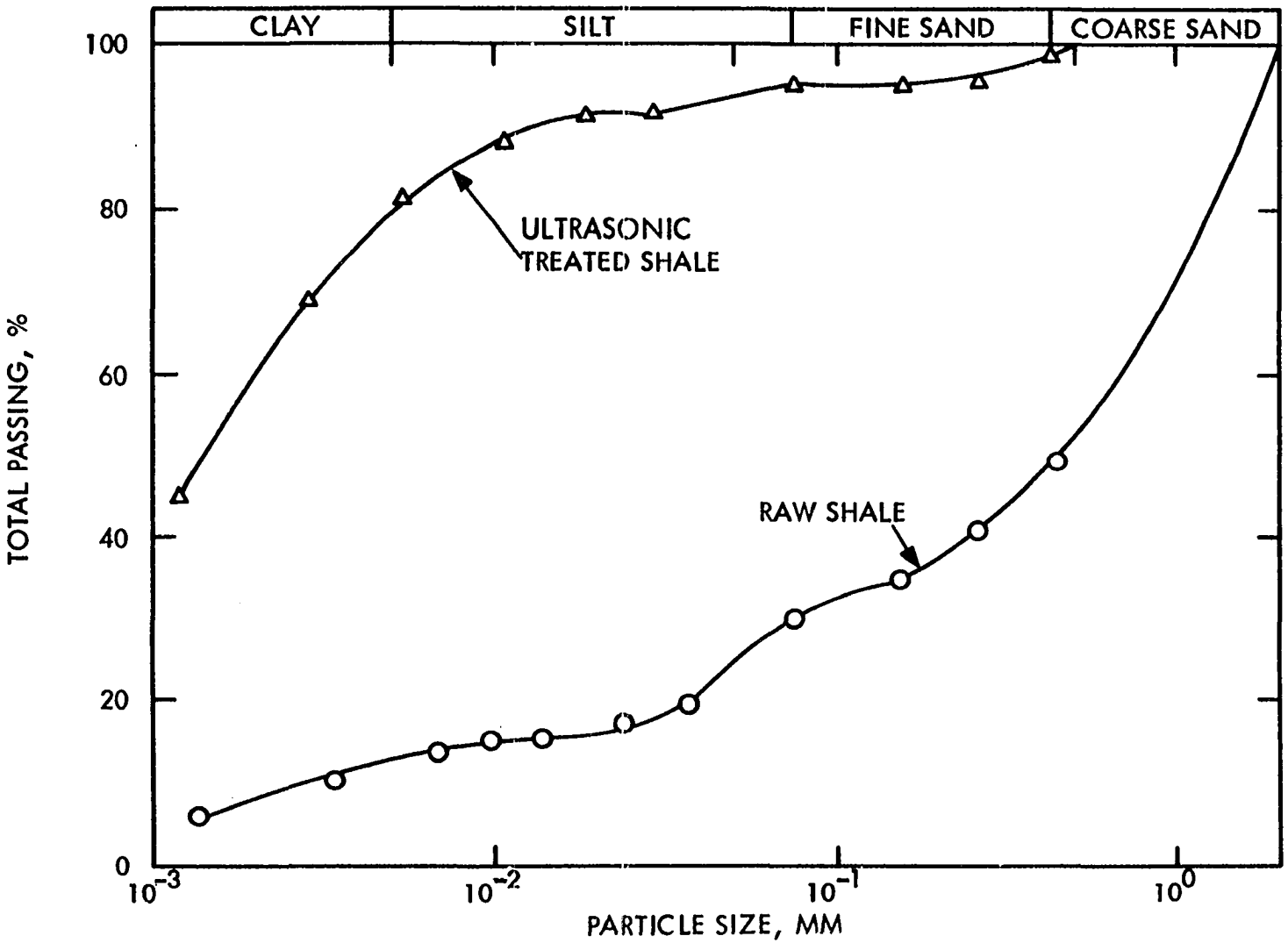


Figure 5.2 Comparison of gradation characteristics for shale 15.

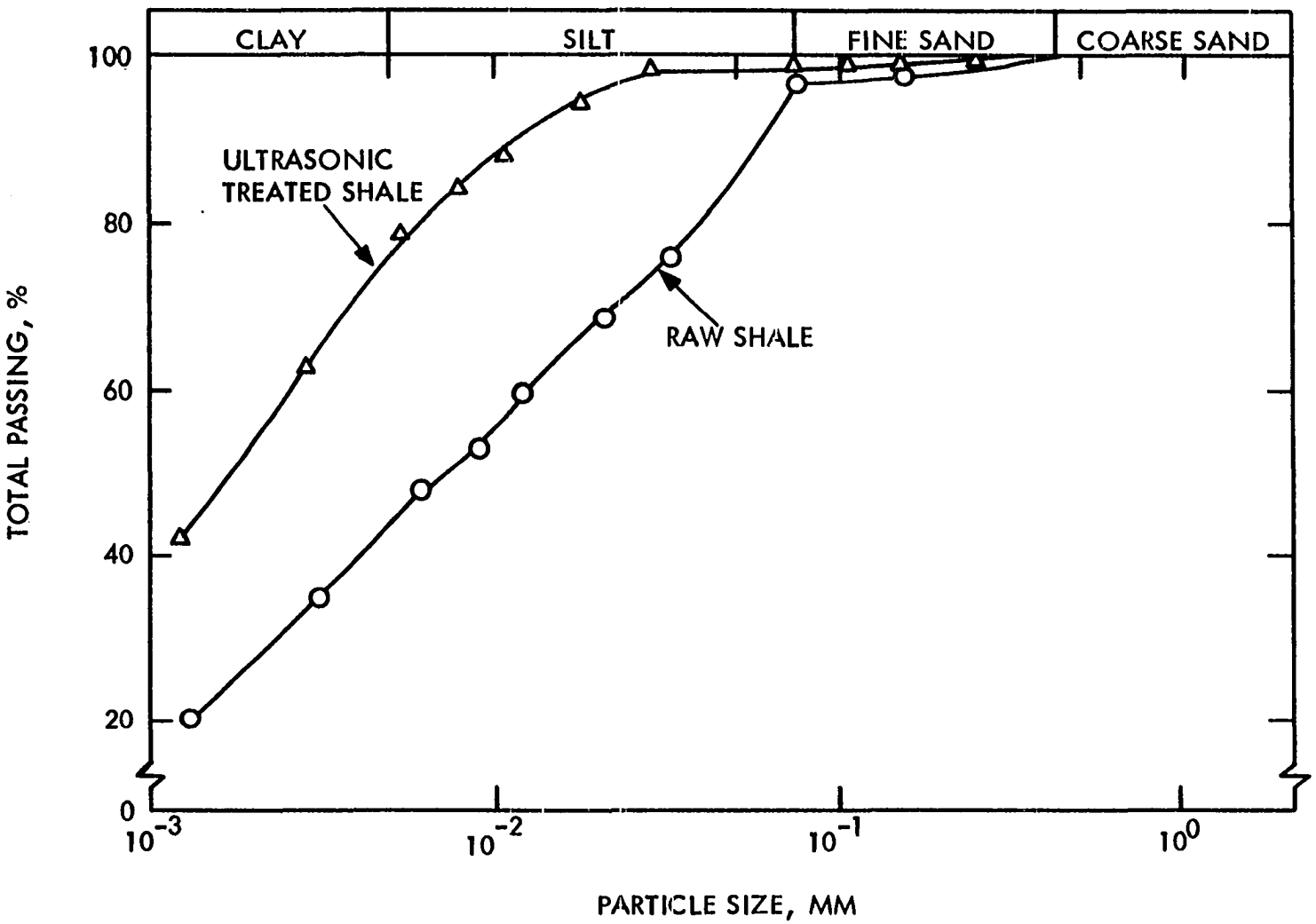


Figure 5.3 Comparison of gradation characteristics for shale 21.

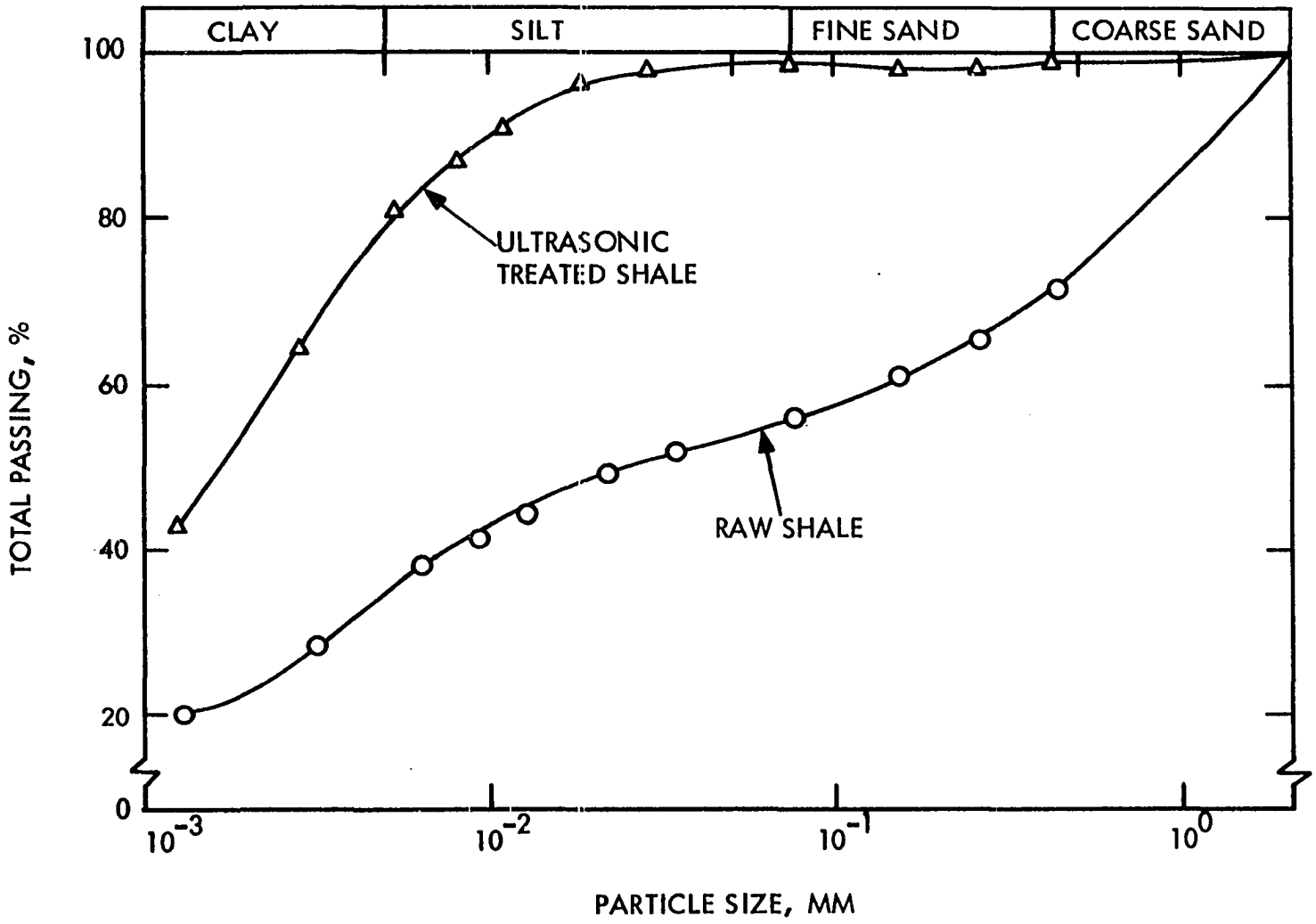


Figure 5.4 Comparison of gradation characteristics for shale 24.

clay and clay size particles are presented in Table 5.1. The gradation for ultrasonic treated shale samples became finer than the gradation for the raw shale samples. However, increases in the amounts of silt and clay seemed to depend on the weathering tendency of the clay shales.

The increase in the amount of 2 micron clay and clay size particles for shale 13 was only 6 percent, being the least among all shales. Shales 15 and 24 appeared very light in color and fine in texture after ultrasonic treatment. The amount of 2 micron clay and clay size particles increased from 9 percent to 60 percent for shale 15 and from 23 percent to 55 percent for shale 24.

These increases showed a similar trend that would be observed in the soils modified by weathering. Chandler (1969, 1972) and Lumb (1965) reported that with weathering the gradations became finer (Figure 5.5). Laguros, Kumar and Annamalai (1974) compared the effects of ultrasonic treatment and natural weathering on some Oklahoma shales and found that the modifications in gradation characteristics caused by both treatments were similar.

Specific Gravity Test Results

As depicted in Table 5.1 the specific gravity of the ultrasonic treated shales decreased slightly within a range of 0.02 to 0.07. No material waste or mineral change was observed as a result of the ultrasonic treatment. Consequently,

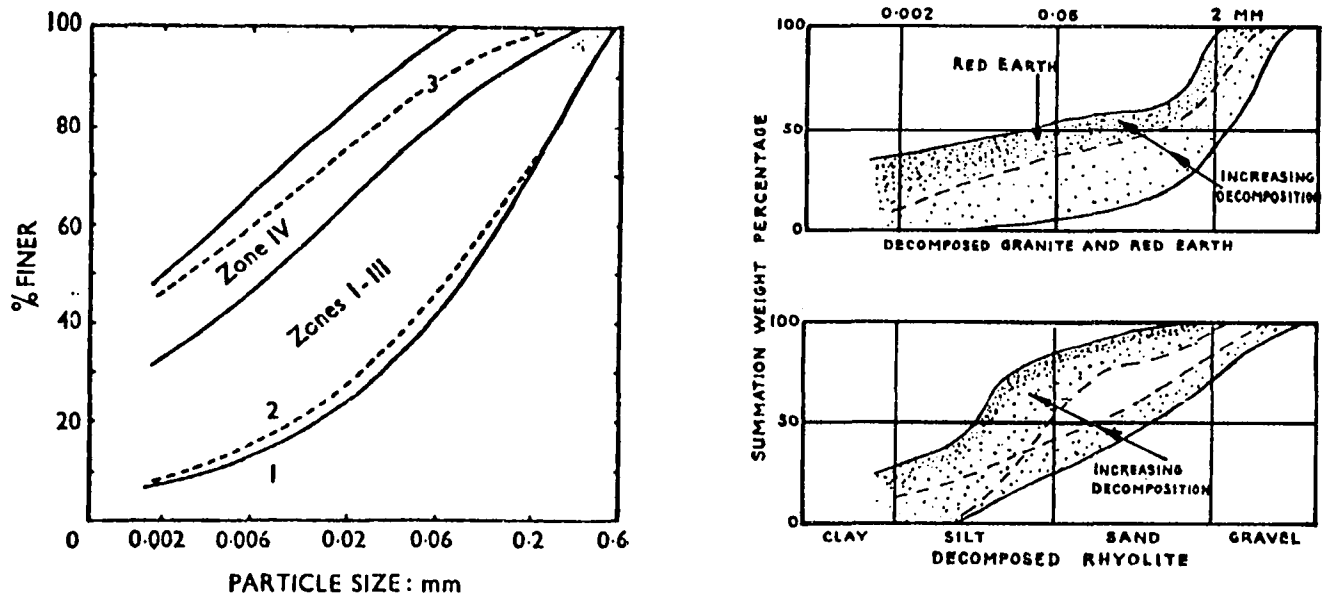


Figure 5.5 Effect of weathering on gradation characteristics of
(a) Hong Kong soils, after Lumb (1965), and
(b) Keuper Marl, after Chandler (1969).

TABLE 5.1

INDEX PROPERTIES OF RAW AND ULTRASONIC TREATED SHALE SAMPLES

Shale No.	Grain Size Analysis			Liquid Limit %	Plastic Limit %	Specific Gravity	Dry Density pcf	Optimum Moisture Content, %
	Silt %	5μ-Clay %	2μ-Clay %					
Raw								
13	32	63	52	47	26	2.73	110.6	18.4
15	18	13	9	33	13	2.77	119.5	13.5
21	53	44	28	29	8	2.79	109.0	19.5
24	21	35	23	38	13	2.73	112.2	17.3
Ultrasonic								
13	32	67	58	51	29	2.70	108.2	19.0
15	15	80	60	45	20	2.70	108.7	20.1
21	23	76	47	33	13	2.76	98.7	24.6
24	11	79	55	47	20	2.71	100.7	23.4

*Amount less than the indicated size.

no change in the values could be expected. However, the difference was small which could be looked at as being due to the normal scatter of the experimental data and not as a result of the treatment.

That the same trend was observed in all the shale samples tested might imply that the small decrease could also be due to the effect of ultrasonic treatment, in which case this difference could only be attributed to the difficulties associated with fine grained soils in determining the specific gravity values. With ultrasonic treatment, the fine grained shale samples become finer and the values of specific gravity of all the shales are similarly affected, hence the trend.

Atterberg Limit Test Results

The liquid limit and plasticity index values are given in Table 5.1. Both the values of liquid limit and plasticity index increased as a result of ultrasonic treatment for all shale samples.

Chandler (1969) reported increases in liquid limit and plasticity index of natural clays with the progress of weathering (Table 5.2). Laguros (1972) observed that these index values also increased with increase in the time of ultrasonic treatment. This trend was quite as expected since any breakdown mechanism, such as natural weathering or ultrasonic treatment or other, causes the breaking of many aggregations of clay and silt particles and releases more of clay

TABLE 5.2

EFFECT OF WEATHERING ON INDEX PROPERTIES OF KEUPER MARL

Index Properties	Weathering Zones *		
	I and II	III	IV
Bulk density (pcf)	140-155	130-145	115-135
Dry density (pcf)	120-150	110-130	85-115
Natural moisture content (%)	5-15	12-20	18-35
Liquid limit (%)	25-35**	25-40	35-60
Plastic limit (%)	17-25**	17-27	17-33
Plasticity index (%)	10-15**	10-18	17-35
% Clay size (B.S. 1377)	10-35	10-35	30-50
Aggregation ratio Ar	10-2.5	10-2.5	2.5

*Zones I, II, III and IV indicate the increasing degree of weathering.

**May be non-plastic.

and clay size fine particles with fresh surfaces. These fine particles change the plasticity characteristics.

Moisture-Density Relationships

The moisture-density relationships for regular and ultrasonic treated shale samples are presented in Figures 5.6 through 5.9. Shales 15, 21 and 24 showed a decrease in density and increase in the amount of optimum water content. Shale 13 also exhibited the same trend but the difference was small. This is probably due to the poor response of this shale to ultrasonic treatment as even the raw shale seemed to be much weathered. The relationship between the values of maximum dry density and optimum moisture content was obtained for 24 shales from various locations in Oklahoma and is shown in Figure 5.10.

The regression equation for the above relationship is

$$MDD = 143.293 - 1.836\ (OMC) \qquad (5.1)$$

where MDD = maximum dry density in pcf

OMC = optimum moisture content in percent

The correlation coefficient for the above relationship is 0.93 and standard error of estimate or root mean square of the deviations about the regression line is 3.34.

The specific gravity values for these shales varied from 2.51 to 2.80, within a narrow range of 0.29. By assuming that the dry density is proportional to the specific gravity

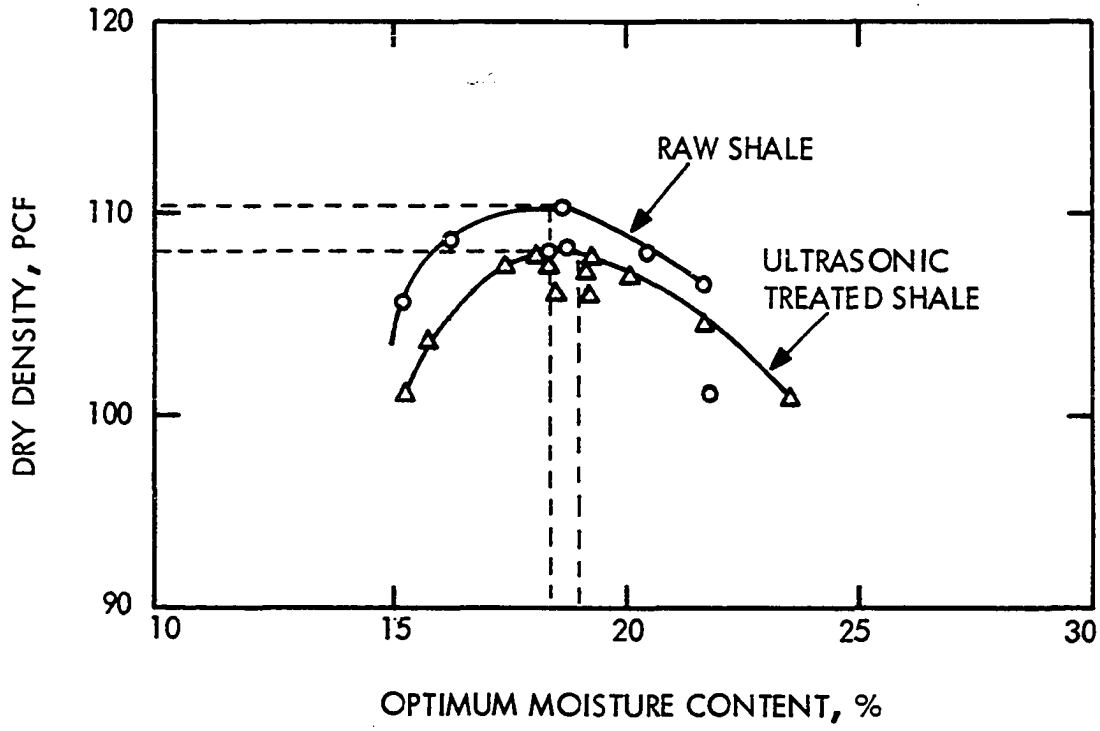


Figure 5.6 Moisture-density relationships for shale 13.

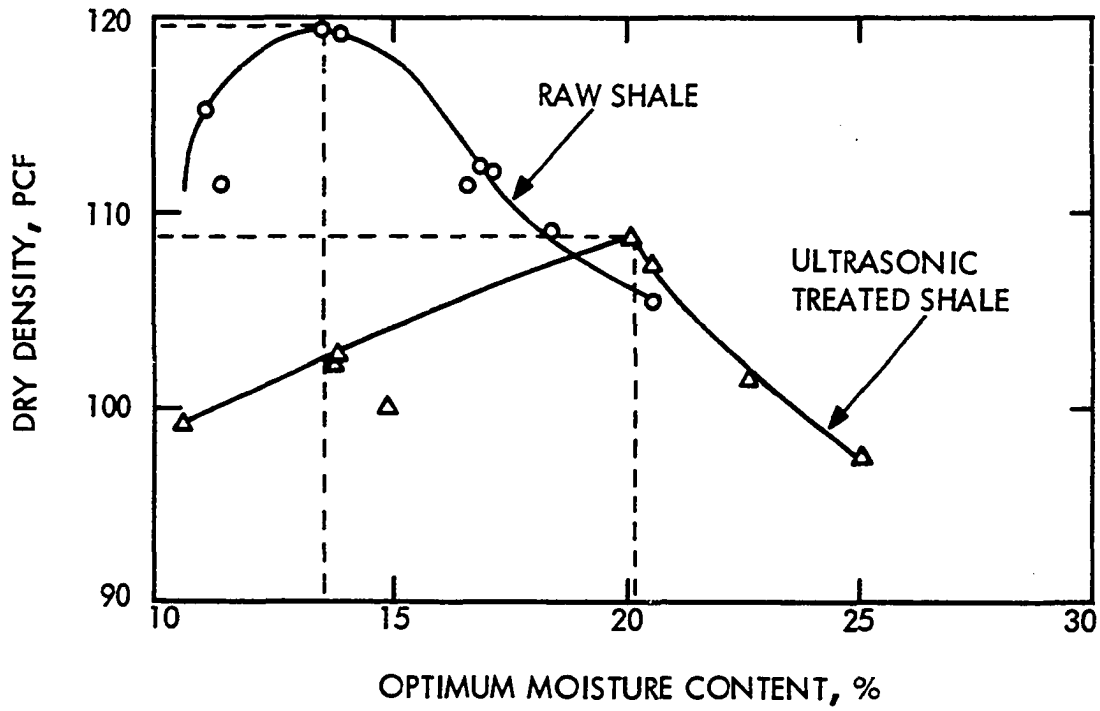


Figure 5.7 Moisture-density relationships for shale 15.

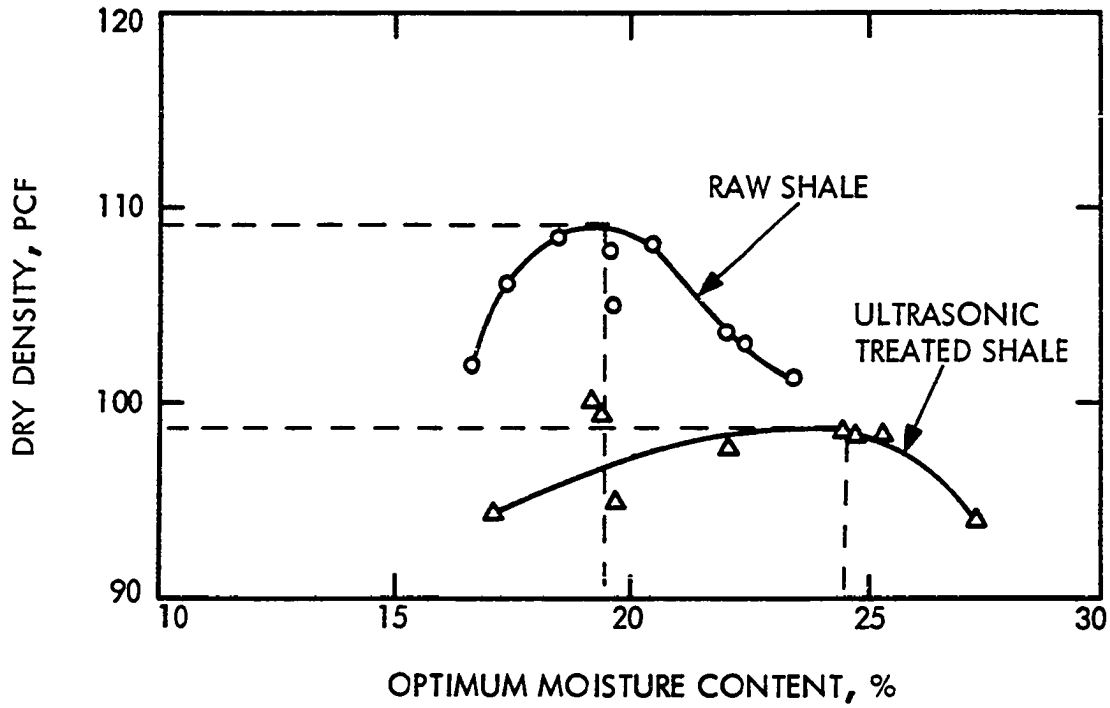


Figure 5.8 Moisture-density relationships for shale 21.

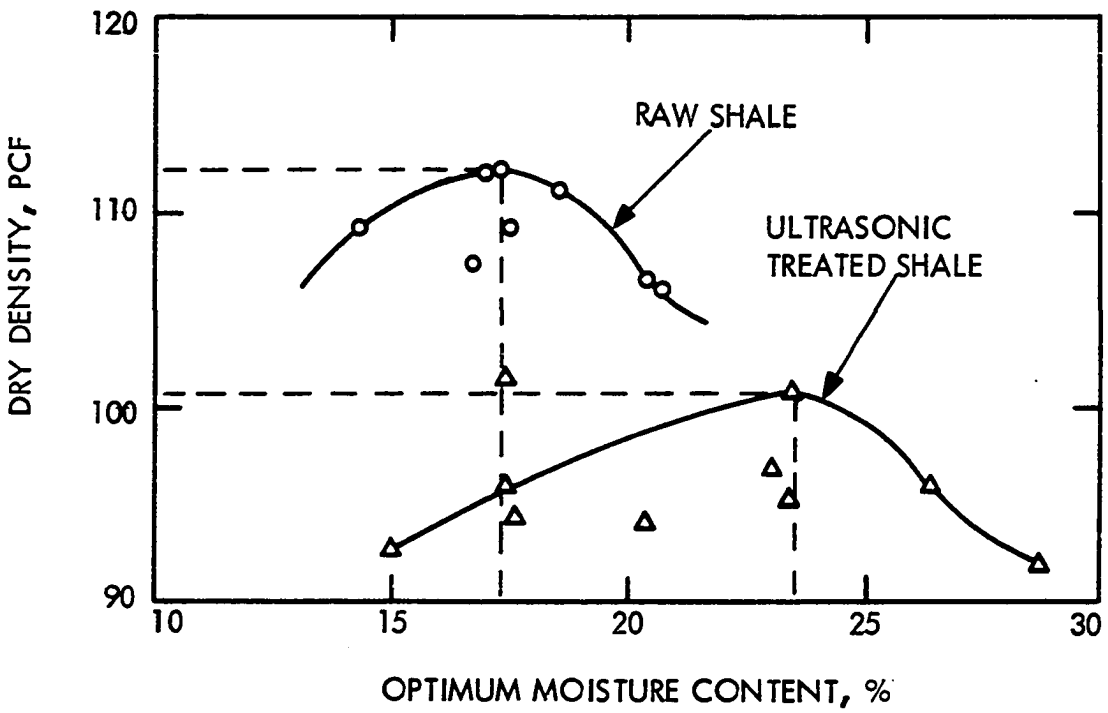


Figure 5.9 Moisture-density relationships for shale 24.

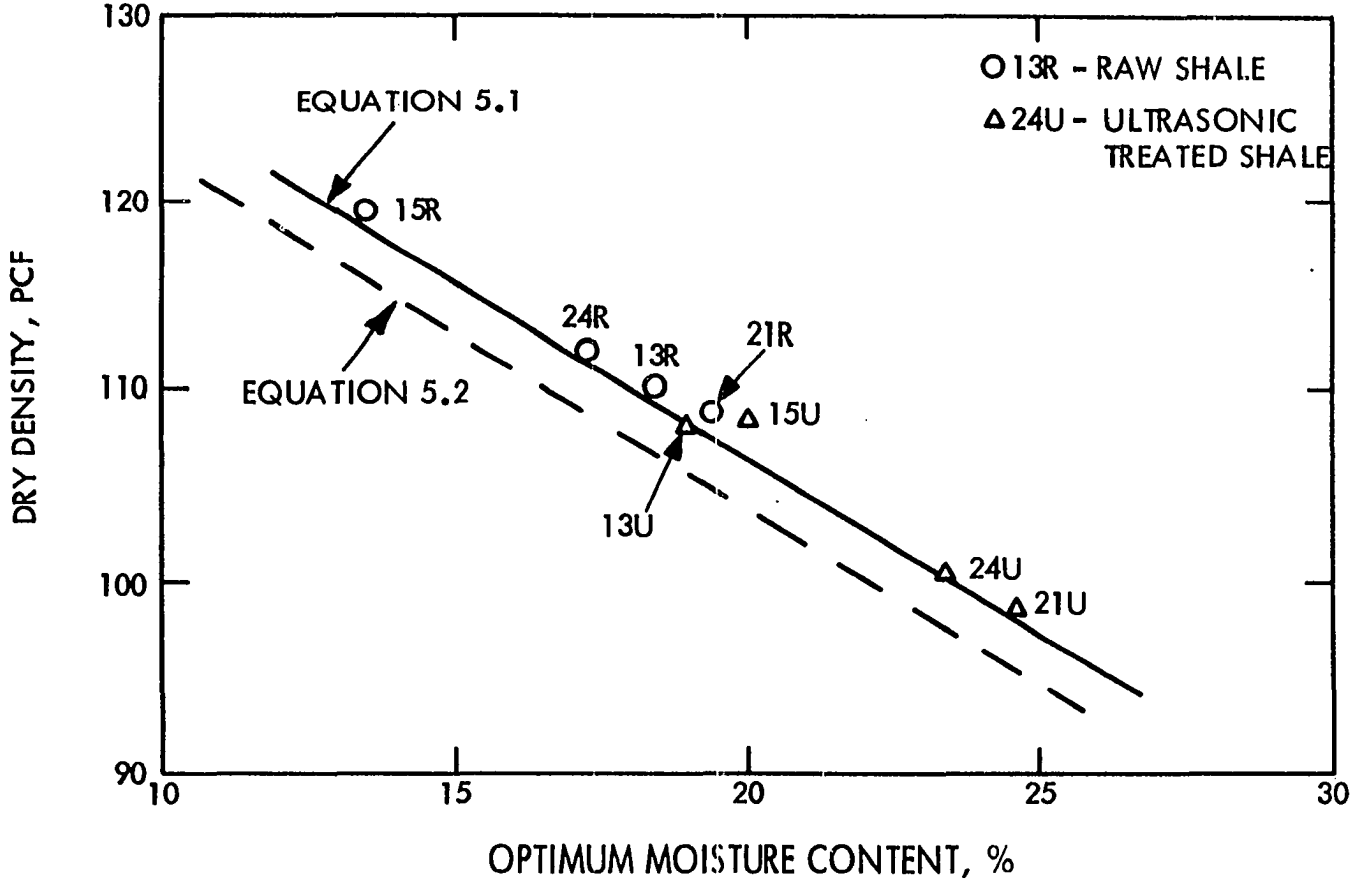


Figure 5.10 Correlation between dry density and optimum moisture content for Oklahoma shales.

of the materials, the values of the dry density are corrected to a hypothetical specific gravity base of 2.65. With the corrected values of dry density and optimum moisture content, the regression equation is

$$MDD_c = 140.820 - 1.845\ (OMC) \qquad (5.2)$$

where MDD_c = MDD (G/2.65), i.e., the maximum dry density values corrected to a hypothetical specific gravity of 2.65, G being the specific gravity of the shale

OMC = optimum moisture content in percent

The correlation coefficient is 0.94 and standard error of estimate is 3.14.

The shales represented at the upper end of the regression line with high densities may be expected to be less weathered and more durable than the shales at the lower end of the line. The moisture-density relationships for the four ultrasonic treated shales studied also follow the same trend as the other untreated shales. After treatment, the density decreased and moisture content increased showing a shift down the regression line. However, the values are interrelated very well by the same regression line. This observation suggests that the breakdown tendency of the shale and the mechanisms involved in ultrasonic treatment are probably similar to those under the influence of natural weathering. The values of dry density for ultrasonic treated samples as predicted by these two equations and as actually determined

are given in Table 5.3. A comparison indicates that those predicted by Equation 5.1 are closer to the actual values than those by Equation 5.2, for all shales in spite of better correlation coefficient of the Equation 5.2.

TABLE 5.3

COMPARISON OF THE PREDICTED VALUES OF DENSITY WITH THE ACTUAL VALUES

Shale Number	Density Actual Values (pcf)	Density[1] (pcf)	Density[2] (pcf)
13	108.20	108.41	107.76
15	108.70	106.39	105.69
21	98.70	98.13	99.39
24	100.70	100.33	99.86

[1]By Equation 5.1.

[2]By Equation 5.2.

Triaxial Compressive Test Results

In the discussion of triaxial compressive test results, the test series designation (Table 4.1) prefixed by shale numbers is used. For example, the series 21R-2 designates raw shale 21 being tested at optimum moisture content and at cell pressures of 0, 10, 20 and 30 psi. In all the triaxial shear tests, the failure was taken as either the actual failure and subsequent decrease in the vertical stress or the condition corresponding to maximum axial deformation of 0.3 in

(10 to 11 percent axial strain). From the stress-strain computations, the maximum deviatoric stress was chosen for analysis.

Control of Specimen Preparation

The desired density and molding water contents were successfully controlled for the majority of the specimens. The variations in molding water content were usually within about 1 percent. The density values varied up to about 7 percent for about 25 percent of the test specimens and 3 percent for the rest. The density variations were found to be more for series 13R-3, 21U-1 and 24U-1 than for other series. The primary reason for these variations is considered to be the operator variability in preparing the samples.

Generally, the variations in density of specimens prepared individually would be more than those prepared from blocks of soil samples. However, blocks of soil samples would require large amount of shale samples. In the case of ultrasonic treated shale samples, this would involve considerable amount of time and prove to be practically difficult. Considering this factor and normal variations in the soil testing, the variations in density and molding water content were considered reasonable.

Strength Data

The most commonly used Mohr-Coulomb equation for the shear strength of soils is

$$\tau = c + \sigma \tan \phi \qquad (5.3)$$

where τ = shear strength, psi

c = cohesion, psi

σ = normal stress, psi

ϕ = angle of internal friction, degrees

The parameters are usually determined from failure envelopes drawn tangentially to the Mohr circles. They are also determined from another simpler method, known as stress path method. If σ_1 and σ_3 are the major and minor principal stresses then the stress path parameters p and q are given by

$$p = (\sigma_1 + \sigma_3)/2 \qquad (5.4)$$

$$q = (\sigma_1 - \sigma_3)/2 \qquad (5.5)$$

The stress path method is found to be very useful in studying the variations in stress conditions during construction and for performance evaluation of the structure.

The failure envelopes obtained from p-q diagrams are referred to as K_f lines or modified Mohr-Coulomb failure envelopes. The conventional shear strength parameters c and ϕ may be calculated from the intercept, K and slope tan α of the modified Mohr-Coulomb failure envelopes by the following relationships:

$$\phi = \sin^{-1} (\tan \alpha) \qquad (5.6)$$

$$c = K/(\cos \phi) \qquad (5.7)$$

The values of dry density, molding water content, maximum deviatoric stress at different lateral pressures, cohesion and angle of friction for raw shales are given in Table 5.4. The above values for ultrasonic treated shales are given in Table 5.5.

Effect of Lateral Pressure

The effect of lateral pressure on stress-strain relationship for raw shale 13 is presented in Figure 5.11. As expected, the lateral pressure, which actually is a confining pressure, caused an increase in the maximum deviatoric stress. With confinement, greater final axial strain could be sustained. However, within the confinement pressure range (10 to 30 psi) no specific trend was observed concerning the final axial strain as there was evidence of some scatter. The stress-strain relationships for ultrasonic treated shales were also similar to the one discussed above. The same pattern was observed for samples compacted at different moisture contents. It agrees well with the typical soil behavior.

Effect of Molding Water Content

The modified Mohr-Coulomb failure envelopes, for raw and ultrasonic treated shales compacted at different molding water contents, are presented in Figures 5.12 through 5.15.

From Table 5.4, for any given shale at a given lateral pressure, it was found that the values of maximum

TABLE 5.4

STRENGTH DATA FOR RAW SHALES

Series Number	Dry Density pcf	Optimum Moisture Content %	Maximum Deviatoric Stress at: $\sigma_3 = 0$ psi	$\sigma_3 = 10$ psi	$\sigma_3 = 20$ psi	$\sigma_3 = 30$ psi	Cohesion psi	Angle of Friction degrees
13R-1	105.7	15.3	49.9	65.2	74.6	83.8	20.0	18.4
13R-2	108.2	18.4	32.9	39.6	43.7	45.9	16.1	7.6
13R-3	100.9	21.8	13.2	18.6	19.2	23.3	6.5	6.0
15R-1	115.3	11.4	40.8	55.3	82.3	92.8	11.5	29.9
15R-2	119.3	13.9	39.3	50.4	56.1	63.1	16.6	15.0
15R-3	112.2	17.1	6.2	8.7	10.4	11.5	3.5	4.0
21R-1	101.9	16.7	35.2	58.9	72.1	80.5	16.6	20.7
21R-2	105.2	19.6	41.1	45.1	53.6	61.7	13.1	17.8
21R-3	103.1	22.4	28.7	31.7	38.2	41.6	10.7	11.7
24R-1	109.1	14.3	51.1	62.5	82.5	98.7	15.2	27.1
24R-2	109.2	17.6	31.8	39.8	47.2	54.3	11.8	16.1
24R-3	106.6	20.4	27.3	30.7	32.1	37.1	11.1	8.6

TABLE 5.5

STRENGTH DATA FOR ULTRASONIC TREATED SHALES

Series Number	Dry Density pcf	Optimum Moisture Content %	Maximum Deviatoric Stress at: $\sigma_3 = 0$ psi	$\sigma_3 = 10$ psi	$\sigma_3 = 20$ psi	$\sigma_3 = 30$ psi	Cohesion psi	Angle of Friction degrees
13U-1	108.2	18.4	41.9	43.9	49.8	53.4	18.2	9.1
13U-2	107.3	19.2	38.6	47.6	52.0	58.0	15.2	14.4
15U-1	102.2	13.5	50.0	72.4	99.8	110.9	14.1	31.4
15U-2	107.3	20.3	34.6	40.2	45.4	50.4	13.8	12.3
21U-1	99.7	19.4	42.6	53.9	68.7	77.3	14.2	23.8
21U-2	98.6	24.6	16.0	28.3	31.3	30.3	9.2	11.2
24U-1	95.9	17.4	48.4	66.4	77.8	96.7	15.1	26.4
24U-2	95.3	23.4	26.0	34.7	50.3	57.1	8.6	21.3

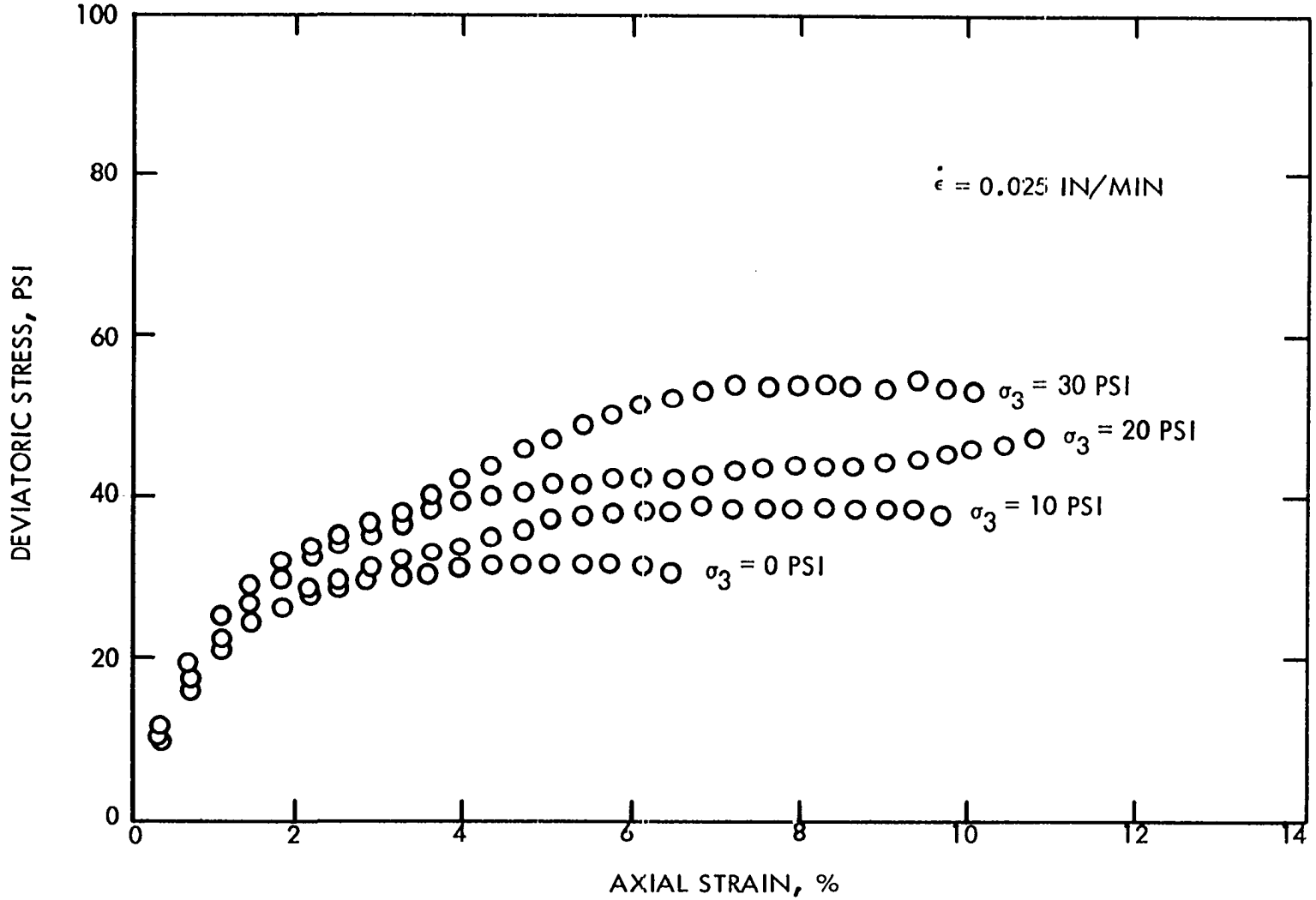


Figure 5.11 Effect of lateral pressure on the shear strength for raw shale 24.

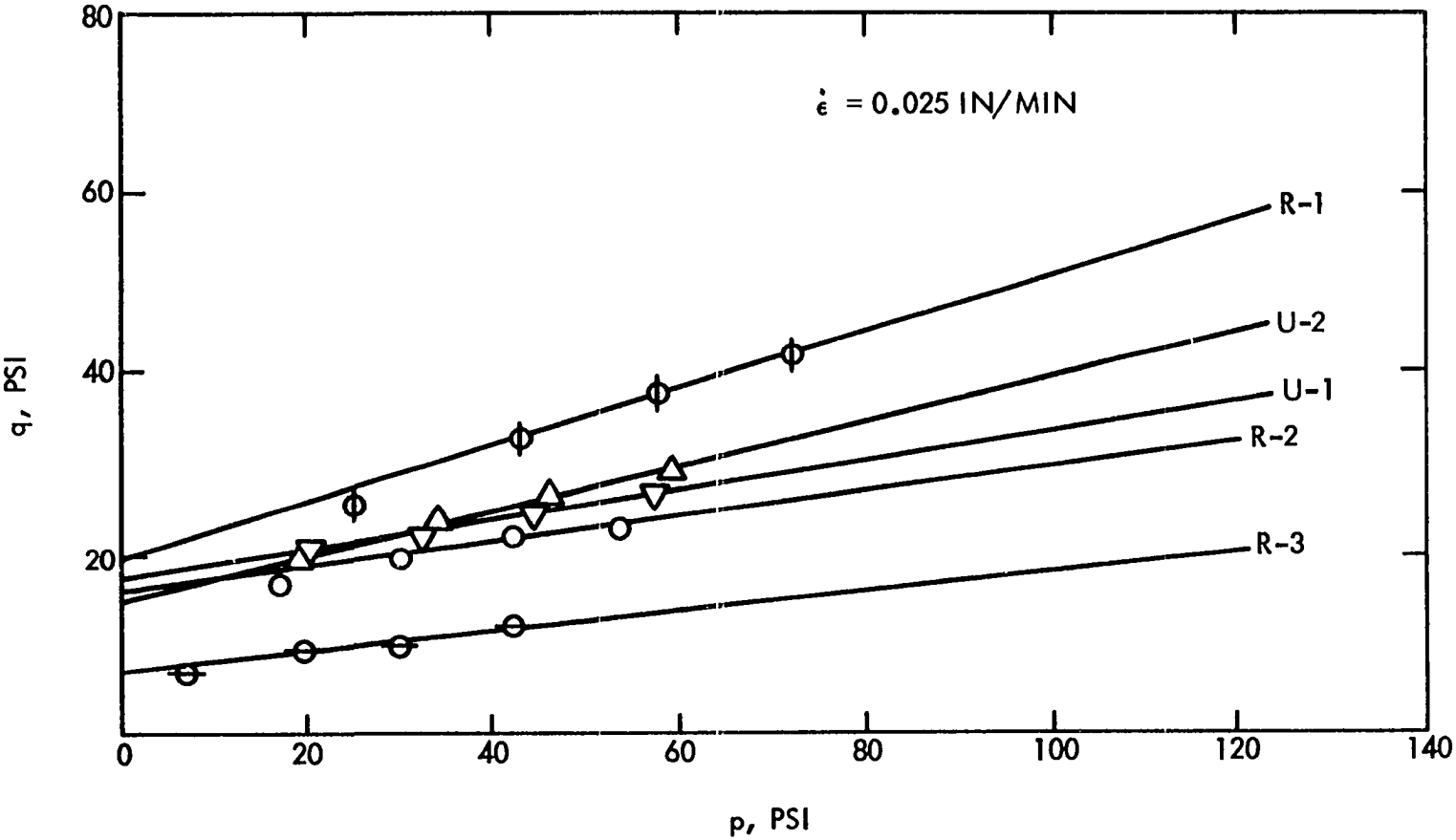


Figure 5.12 Modified Mohr-Coulomb failure envelopes for shale 13.

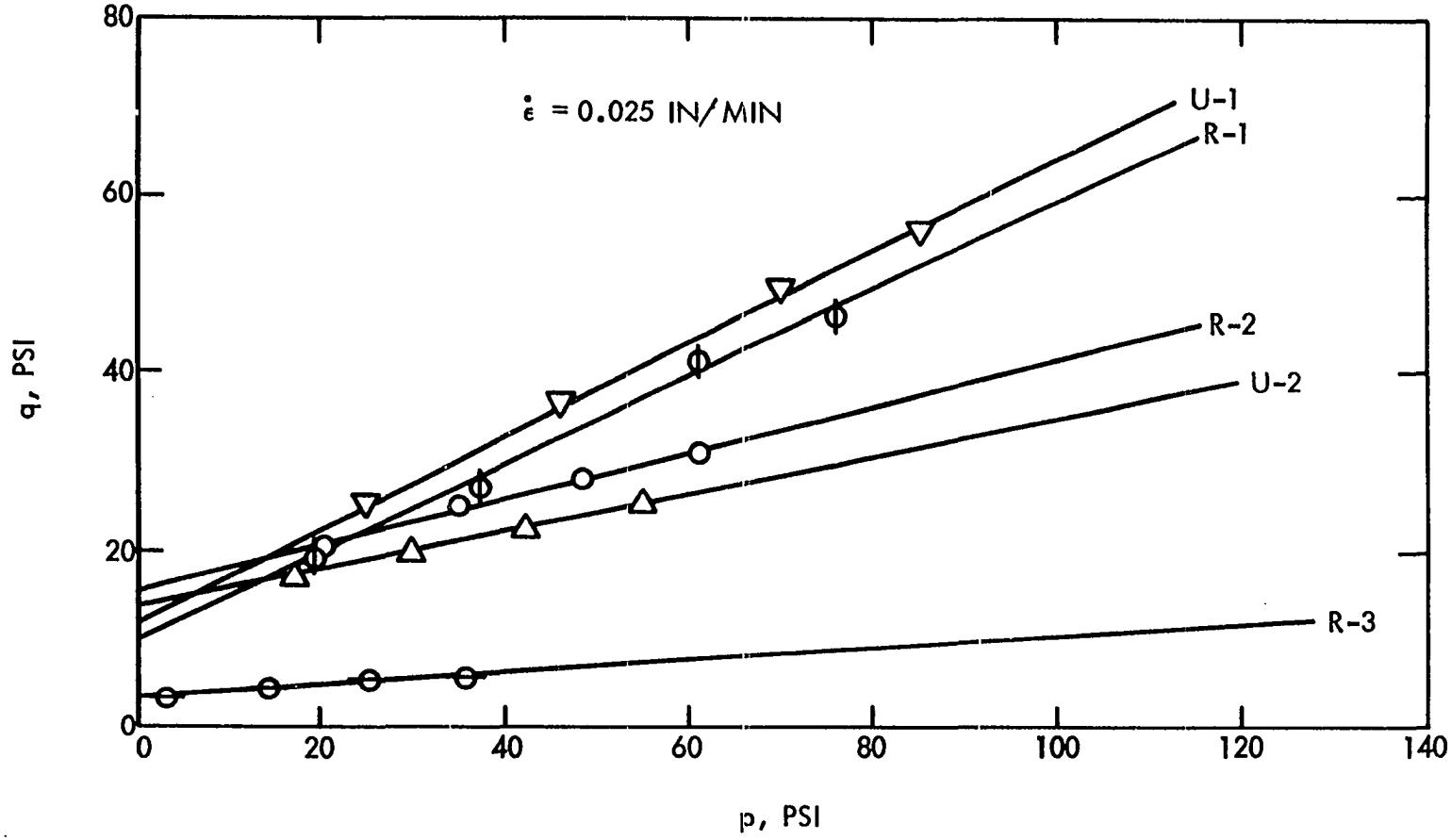


Figure 5.13 Modified Mohr-Coulomb failure envelopes for shale 15.

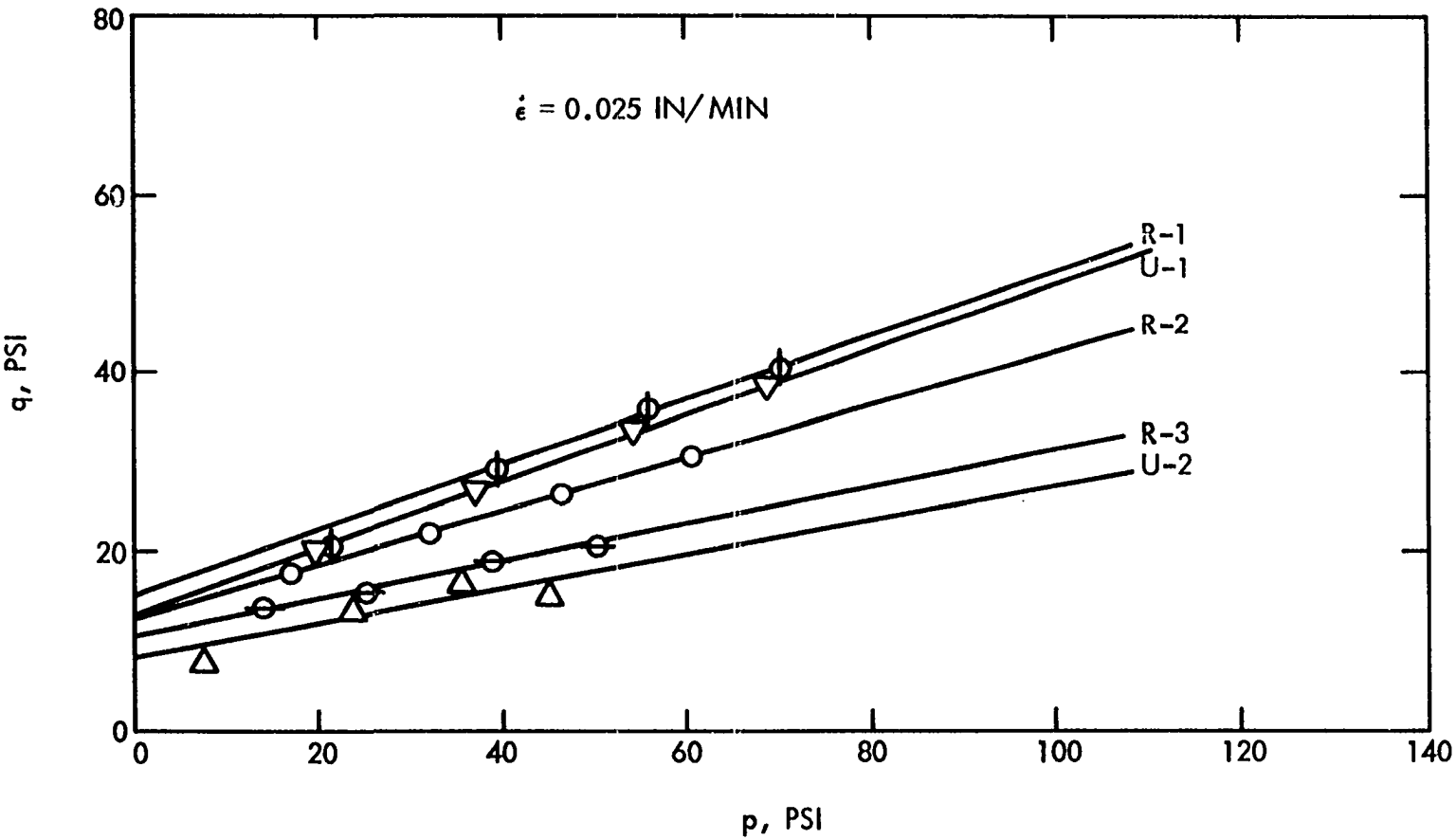


Figure 5.14 Modified Mohr-Coulomb failure envelopes for shale 21.

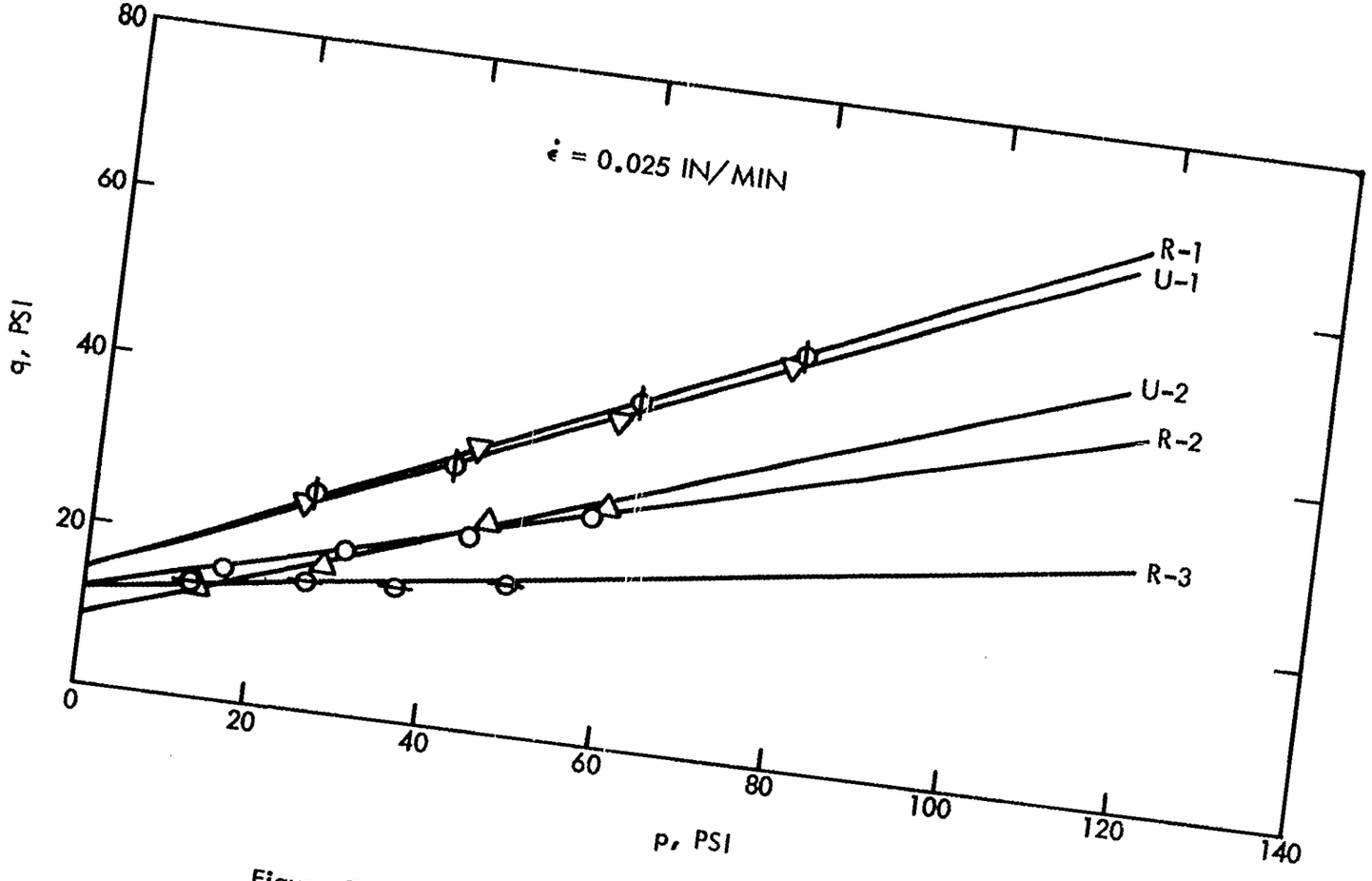


Figure 5.15 Modified Mohr-Coulomb failure envelopes for shale 24.

deviatoric stress decreased with increase in molding water content. Among these shales, shale 15 showed high strength loss when the moisture content was increased by 3 percent over the optimum moisture content. For example, at a lateral confinement pressure of 30 psi, the maximum deviatoric stress decreased from 63.1 psi to 11.5 psi with increase in moisture content. The following explanation is offered for this observation.

Shale 15 contained about 87 percent of silt and sand combined and consequently had high density and low optimum moisture content. In the undisturbed state, it is classified as a good construction material (Oklahoma Department of Highways, 1966). However, it seemed that in the laboratory much of the interparticle bonds were destroyed in the process of grinding and mixing. Later, some of the interparticle bonds might have been reestablished, in the presence of thin film of water around the particles, by compaction processes.

Yet the recovery of interparticle bonds during compaction seemed to be a function of size, shape and mineralogical characteristics of the constituent particles. During mixing and handling, it was observed that shale 15 changed quite differently in its appearance and stability after compaction with changes in molding water contents. At water contents, on the wet side of optimum, the specimens looked very wet and the strength was correspondingly low.

In series 13R-3, it was found very difficult to obtain specimens of desired density as the shale samples formed very hard clay lumps with increase in moisture content and included many air pockets in the compacted specimens. Generally for samples compacted at wet of optimum, density would be low but saturation would be high. Because of air pockets, saturation was low for this series. It increased from 89.9 percent to 90.1 percent for a 3 percent increase in moisture content. Also the density and strength were low.

From the Figures 5.12, 5.13, 5.14 and 5.15, it can be seen that the K_f line for series compacted dry of optimum (R-1) lie above the K_f lines for series compacted at optimum (R-2) and wet of optimum (R-3). The values of cohesion and angle of friction also decreased with increase in molding water content (Table 5.4). Among the ultrasonic treated shales, the series U-1 refers to the samples compacted at optimum moisture content determined for raw shale. The series U-2 refers to the samples compacted at optimum moisture content determined for ultrasonic treated shale and hence the molding water content is more than that for series U-1.

For ultrasonic treated shales 15, 21 and 24, the maximum deviatoric stress decreased with increase in moisture content. However, shale 13 did not exhibit this behavior as the difference in molding water content for series U-1 and U-2 was only 0.8 percent (molding water content for U-1 series is 18.4 percent and for U-2 series, 19.2 percent).

Also the deviatoric values changed inconsistently for different lateral pressures. At σ_3 = 0 psi, the deviatoric stress decreased from 41.9 psi to 38.6 psi with increase in moisture content. At other lateral pressures, the reverse trend was noticed. The differences in the values of maximum deviatoric stress were also less, being of the order of 2.2 psi to 4.6 psi. This could be due to the normal variations in molding water content and density values among the series the molding water contents of which were separated only by a narrow margin of 0.8 percent.

Effect of Ultrasonic Treatment

After ultrasonic treatment, the density decreased and optimum moisture content increased. Consequently, a lower strength would be expected. This was the trend seen from the modified Mohr-Coulomb failure envelopes for the raw and ultrasonic treated shale samples compacted at their respective moisture contents (series R-2 and U-2) which are shown in Figures 5.12 through 5.15. For shales 15 and 21 the strength envelope for ultrasonic treated shale was lower than that for raw shales. For shales 13 and 24, the strength decrease was not obvious.

Regarding shale 13, the effect of ultrasonic treatment on shale was generally found to be less as indicated by gradation and plasticity tests. Also the molding water content varied only slightly (from 18.4 percent to 19.2 percent).

For shale 24, the maximum deviatoric stress for raw shale was more than that for ultrasonic treated shale at lateral confinement pressures of 0 psi and 10 psi. But at 20 psi and 30 psi, the ultrasonic treated shale had higher maximum deviatoric stress than the raw shale. The increases were 3.1 psi at σ_3 = 20 psi and 2.8 psi at σ_3 = 30 psi. Since only the total stress was measured, such a small scattering could be due to the variations in pore water movement and incomplete dissipation of pore water pressure at the rate of strain tested.

To verify this, a slow rate of strain of 0.0025 in/min was used and the series R-2, U-1 and U-2 were repeated. These are designated as R-2(S), U-1(S) and U-2(S). The strength data values are given in Table 5.6. The modified Mohr-Coulumb failure envelopes for the series R-2(S), U-1(S) and U-2(S) are presented in Figures 5.16, 5.17, 5.18 and 5.19.

At this rate of strain, the strength of raw shale was found to be higher than that for ultrasonic treated shale for all shales except shale 13. The failure envelopes for all three series were almost the same for this shale probably for the same reason that the shale 13 changed only slightly on ultrasonic treatment.

Shale 24, at the low rate of strain, indicated the same pattern as shales 15 and 21 showing decrease in strength after ultrasonic treatment. Hence the discrepancy observed for shale 24 at the strain rate of 0.025 in/min seemed to be due to variations in the pore water movement.

TABLE 5.6

STRENGTH DATA AT STRAIN RATE OF 0.0025 IN/MIN

Shale No.	Series Desig.	Dry Density pcf	Optimum Moisture Content %	Maximum Deviatoric Stress at: σ_3=0 psi	σ_3=10 psi	σ_3=20 psi	σ_3=30 psi	Cohesion psi	Angle of Friction degrees
13	R-2(S)	108.5	18.8	30.2	39.1	46.9	49.8	13.8	12.3
13	U-1(S)	106.2	18.6	38.8	45.0	48.5	50.7	20.2	8.1
13	U-2(S)	106.2	19.2	37.6	39.5	41.0	48.8	15.7	9.1
15	R-2(S)	120.9	13.8	19.0	43.0	48.1	58.7	8.6	21.3
15	U-1(S)	102.6	13.7	47.8	76.5	92.9	113.4	15.1	30.6
15	U-2(S)	108.6	20.1	24.2	26.3	29.9	33.8	10.6	7.6
21	R-2(S)	107.9	19.6	44.3	51.2	56.8	63.0	18.4	22.6
21	U-1(S)	100.4	19.2	56.3	70.7	81.3	90.0	19.5	21.3
21	U-2(S)	98.4	24.8	26.7	31.9	33.7	35.2	14.5	3.5
24	R-2(S)	112.2	17.3	39.2	53.1	59.8	68.9	17.2	16.1
24	U-1(S)	101.5	17.5	59.4	74.0	87.7	104.1	18.8	25.1
24	U-2(S)	100.2	23.5	29.2	36.1	41.7	46.2	11.7	12.8

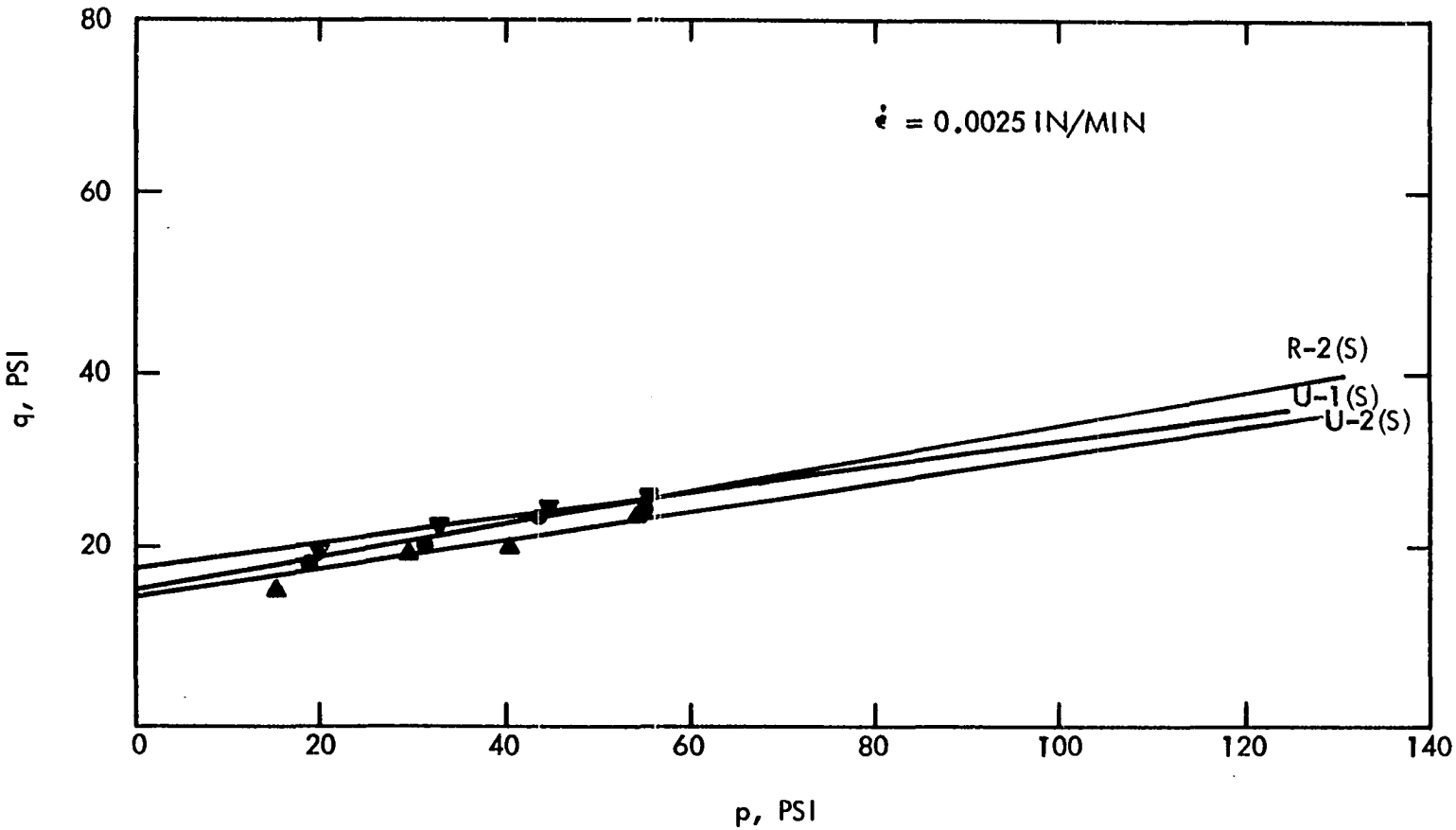


Figure 5.16 Modified Mohr-Coulomb failure envelopes for shale 13.

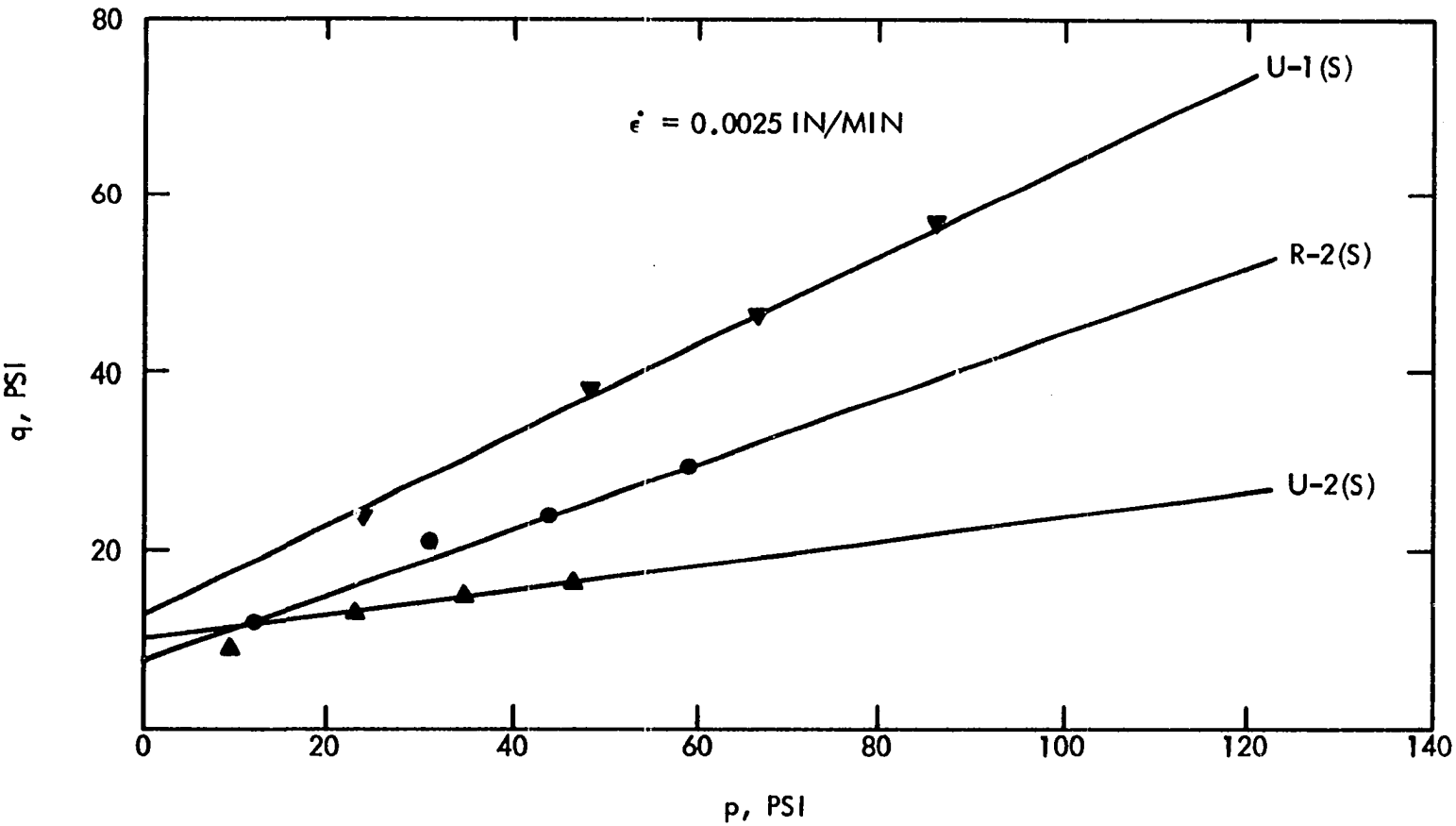


Figure 5.17 Modified Mohr-Coulomb failure envelopes for shale 15.

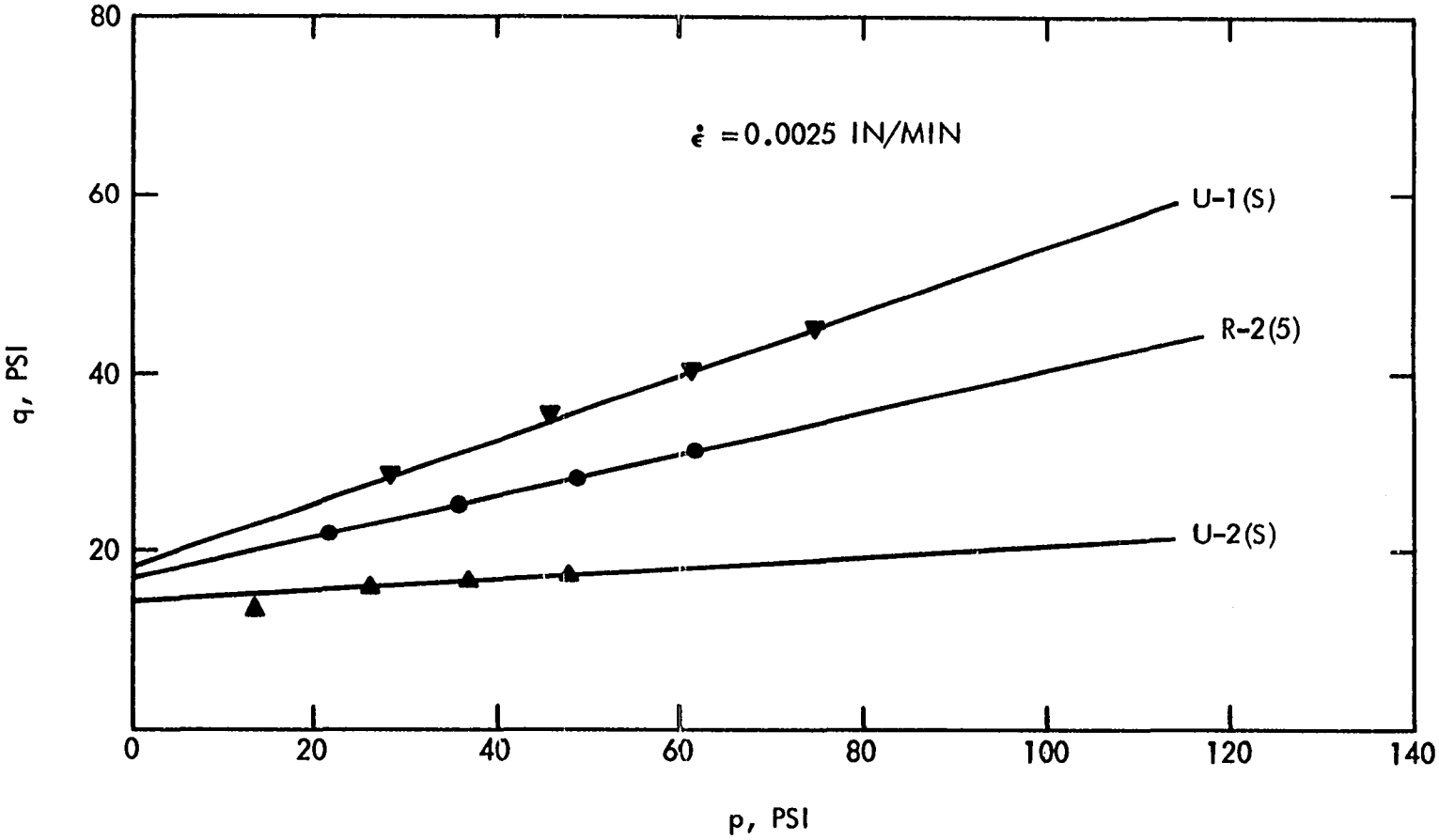


Figure 5.18 Modified Mohr-Coulomb failure envelopes for shale 21.

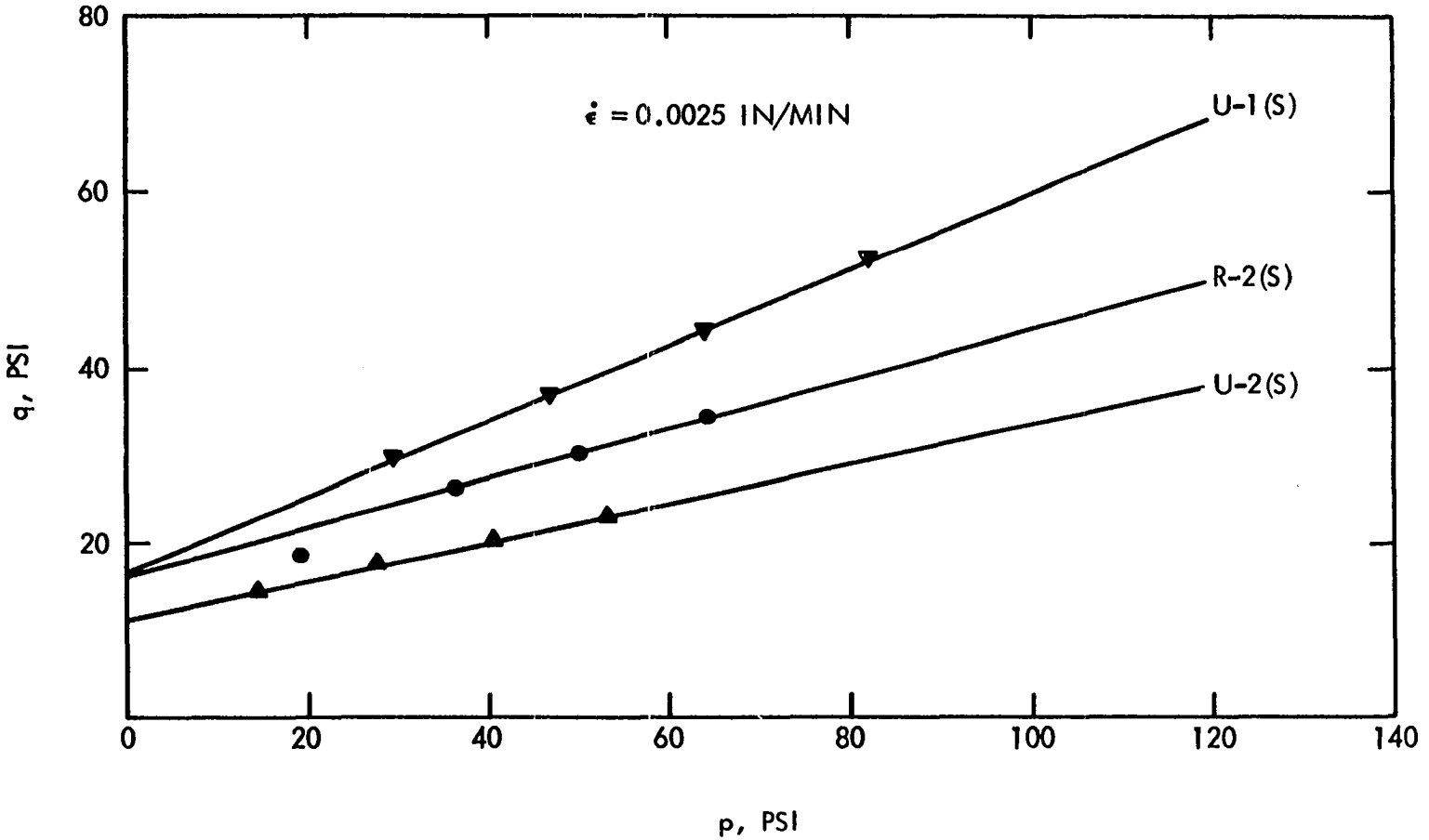


Figure 5.19 Modified Mohr-Coulomb failure envelopes for shale 24.

On comparison of the raw and ultrasonic treated shales, compacted at the same water content, i.e., at the optimum moisture content determined for raw shales (series R-2 and U-1), it was found that the maximum deviatoric stress for ultrasonic treated shale increased from that for raw shale. Also from Figures 5.16 through 5.19, the failure envelopes indicated that at the same molding water content, the strength for ultrasonic treated shale was more than that for raw shale (K_f line for U-1 lying above that for R-2). The same trend was observed for tests performed at slow rate of strain of 0.0025 in/min (K_f line for U-1(S) lying above that for R-2(S)).

However, the reason for higher strength of ultrasonic treated shale (compared at the same water content) was probably that the treated shale sample produced a dried mix than the raw shales at the same water content. This resulted in the formation of aggregations or lumps of clay size particles and manifested higher strength.

Similar observations were reported in the case of naturally weathered soils (Chandler, 1972). Increases in field moisture contents with weathering were noticed. The zones of weathering were identified by determining the oxidation ratio or the ratio of Fe_2O_3/FeO which increased with weathering. Figure 5.20 shows the progressive changes in the relationship between strength and moisture content with weathering. The shear strength of weathered soils in

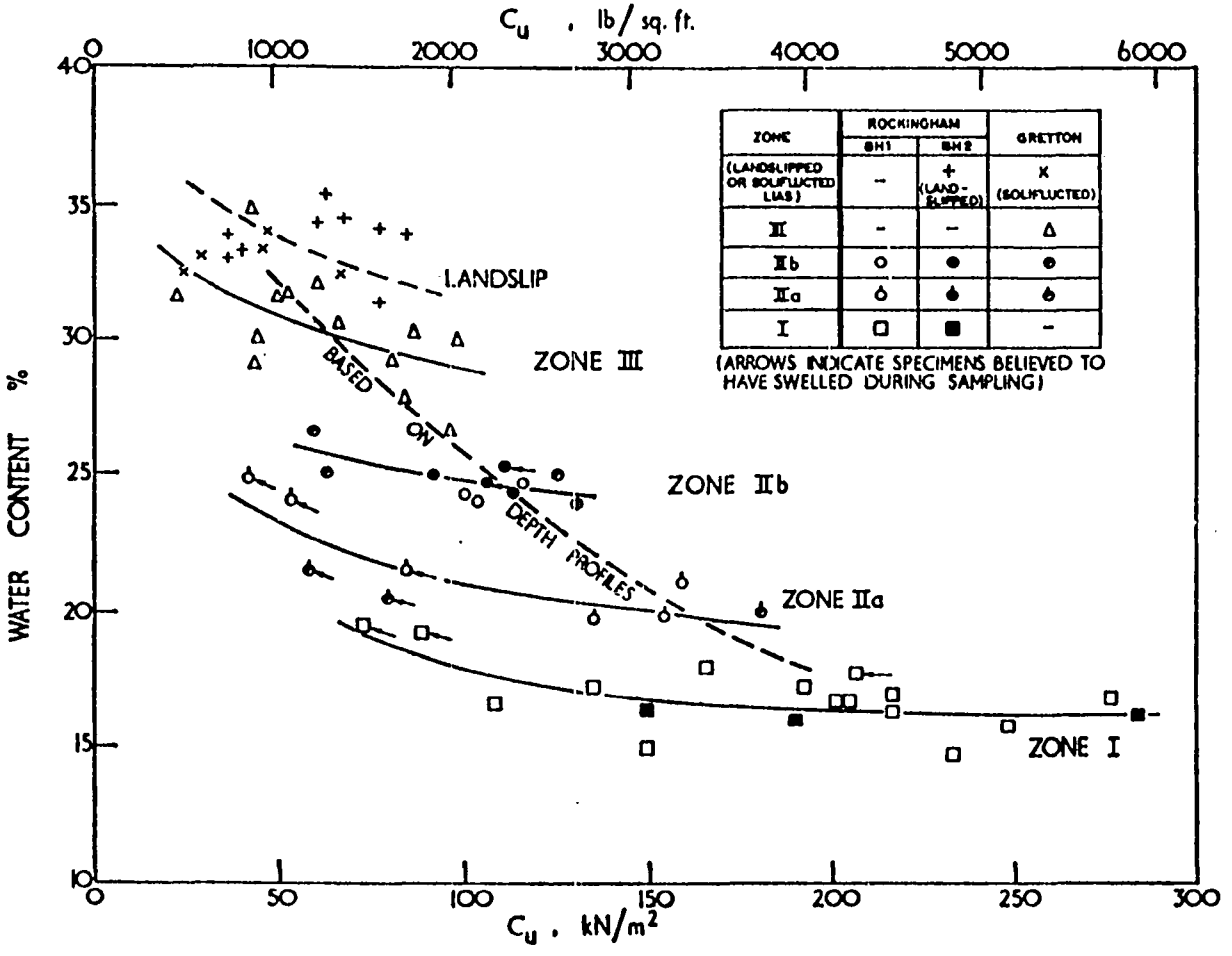


Figure 5.20 Effect of weathering on shear strength - water content relationships for Lisa clay, after Chandler (1972).

undisturbed condition was lower than that of unweathered soils. However, at a given water content, weathered soils showed higher strength.

Figure 5.21 shows the relationship between unconfined compressive strength and moisture content before and after ultrasonic treatment for shale 24. The unconfined compressive strength decreased as a consequence of ultrasonic treatment for samples compacted at their respective moisture content. But at the same water content, the unconfined compressive strength for ultrasonic treated shale was higher than that for raw shale. The comparison of these relationships substantiates the thesis that ultrasonic treatment simulated natural weathering in relation to the strength properties.

Modes of Failures

An examination of failed specimens indicated that only three distinct types of failures occurred. They seemed to be governed by molding water content and degree of lateral confinement. As the same patterns were observed for both raw and ultrasonic treated shales, the effect of ultrasonic treatment did not appear to influence the mode of failure.

The general modes of failure are shown in Figure 5.22 and along with their influencing factors are briefly summarized below.

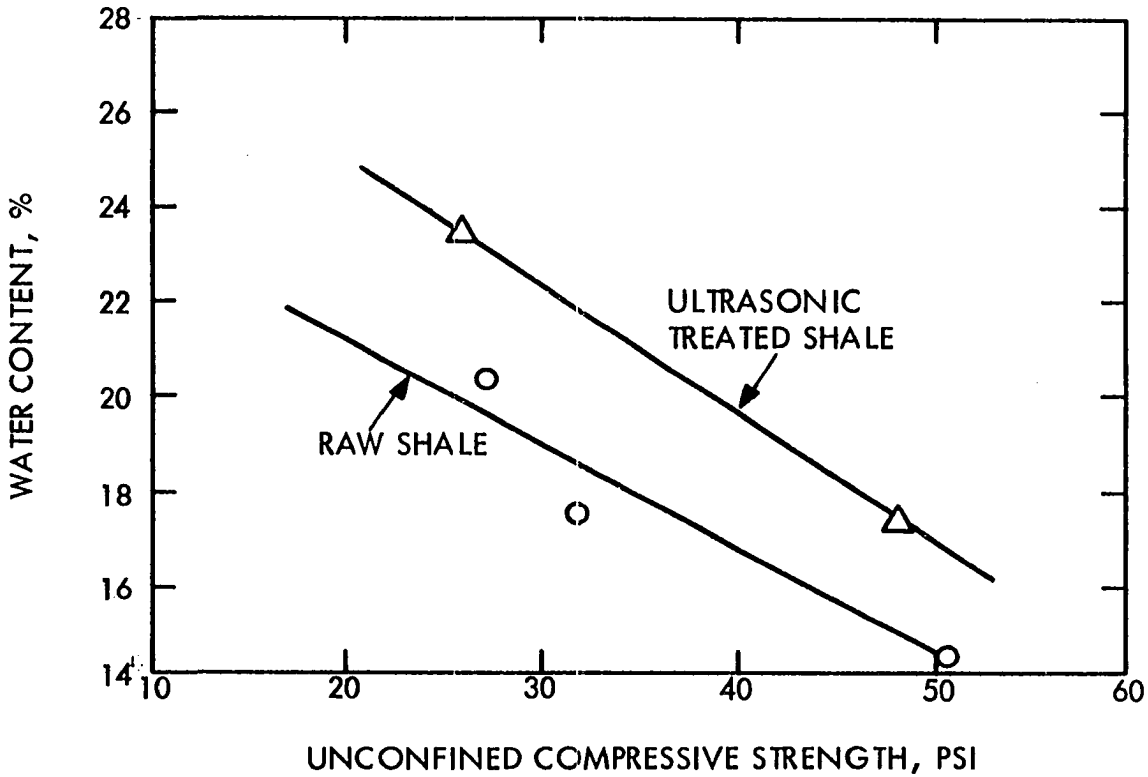


Figure 5.21 Effect of ultrasonic treatment on shear strength-water content relationship for shale 24.

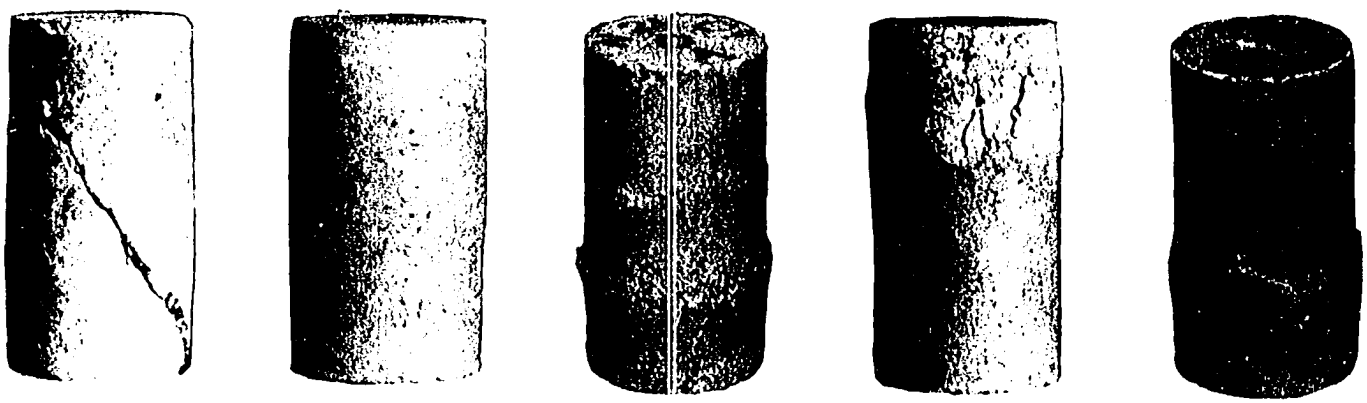


Figure 5.22 Failure patterns of specimens.

Shear failures: Figure 5.22a shows a typical shear failure which generally occurred with well defined single shear plane. This type of failure was found to occur in shale samples (both raw and ultrasonic treated) compacted at molding water content less than optimum and tested under low confining pressures. The stress-strain curve showed a "peak stress." On the dry side of optimum, the structure was turbostratic in microscopic scale and contained large aggregations of clay sized particles. The failure occurred was typical of frictional materials with well defined failure plane.

Compression failure: Shown in Figure 5.22b is a typical compression failure which was observed in the case of specimens compacted at molding water contents more than optimum. Well defined shear planes were absent. These specimens deformed uniformly in vertical direction and radial deformations were also more or less uniform throughout the height of the specimens. The stress-strain relationships did not indicate a peak stress and hence maximum failure stress for these specimens was taken as the stress at the strain corresponding to 0.30 in of deformation (10 percent to 11 percent).

Multiple shear failure: Most of the specimens failed exhibiting this mode of failure as shown in Figures 5.22c, d and e. Failure seemed to develop along more than one plane,

mostly restricted within a small zone. But the zone containing the failure planes occurred anywhere along the height of the specimens, sometimes near the ends and other times near the midheight of the specimens. The shifting of position of the zone of multiple shear planes might be due to the variation of density of the specimen along the vertical direction and the shear planes started to develop at the weakest zones.

Relaxation Test Results

In relaxation tests, the only variables used are the rates of strain application, lateral pressure and ultrasonic treatment. The values of deviatoric stress at the beginning and the end of the relaxation for both ultrasonic treated and raw shales are presented in Table 5.7. Also typical patterns are presented in Figures 5.23, 5.24 and 5.25 to illustrate the effect of the above variables on the relaxation characteristics. Figure 5.23 shows the effect of lateral pressure on the relaxation properties of ultrasonic treated shale 15. The deviatoric stress at 50 percent of failure strain and just before relaxation increased from 16.2 psi at σ_3 = 10 psi to 23.1 psi at σ_3 = 30 psi. The same pattern was observed for raw shales and also at low strain rate.

Figure 5.24 shows the effect of ultrasonic treatment on the relaxation characteristics for shale 24. Since both the raw and ultrasonic treated shales were compacted at their respective optimum moisture contents, deviatoric stresses for

TABLE 5.7

VALUES OF DEVIATORIC STRESS AT THE BEGINNING AND END OF RELAXATION

Shale Number*	Speed in/min	Deviatoric Stress (psi) at: σ_3 = 10 psi		σ_3 = 20 psi		σ_3 = 30 psi	
		Begin	End	Begin	End	Begin	End
13R	0.025	28.6	20.6	36.2	31.5	38.8	33.8
13U	0.025	38.1	32.0	39.6	31.9	46.1	38.7
13R	0.0025	35.4	33.0	38.6	36.3	41.1	38.4
13U	0.0025	37.6	34.0	40.9	37.3	45.2	41.3
15R	0.025	39.3	29.9	41.1	32.6	49.4	40.5
15U	0.025	16.2	11.5	20.3	16.1	23.1	19.2
15R	0.0025	25.8	24.3	32.1	29.3	37.4	34.6
15U	0.0025	18.5	17.3	20.0	18.5	22.0	20.9
21R	0.025	42.5	32.7	45.7	36.5	50.3	39.2
21U	0.025	22.4	14.8	25.7	18.3	30.6	24.6
21R	0.0025	48.2	43.2	56.7	51.1	59.7	54.0
21U	0.0025	28.8	26.8	30.8	28.2	31.8	29.4
24R	0.025	45.7	37.5	51.7	42.5	56.2	47.5
24U	0.025	36.9	29.3	45.2	38.1	45.0	38.1
24R	0.0025	45.7	41.7	52.3	47.5	55.1	52.1
24U	0.0025	29.5	27.3	34.8	32.6	39.1	36.0

*R - raw shale, and U - ultrasonic treated shale.

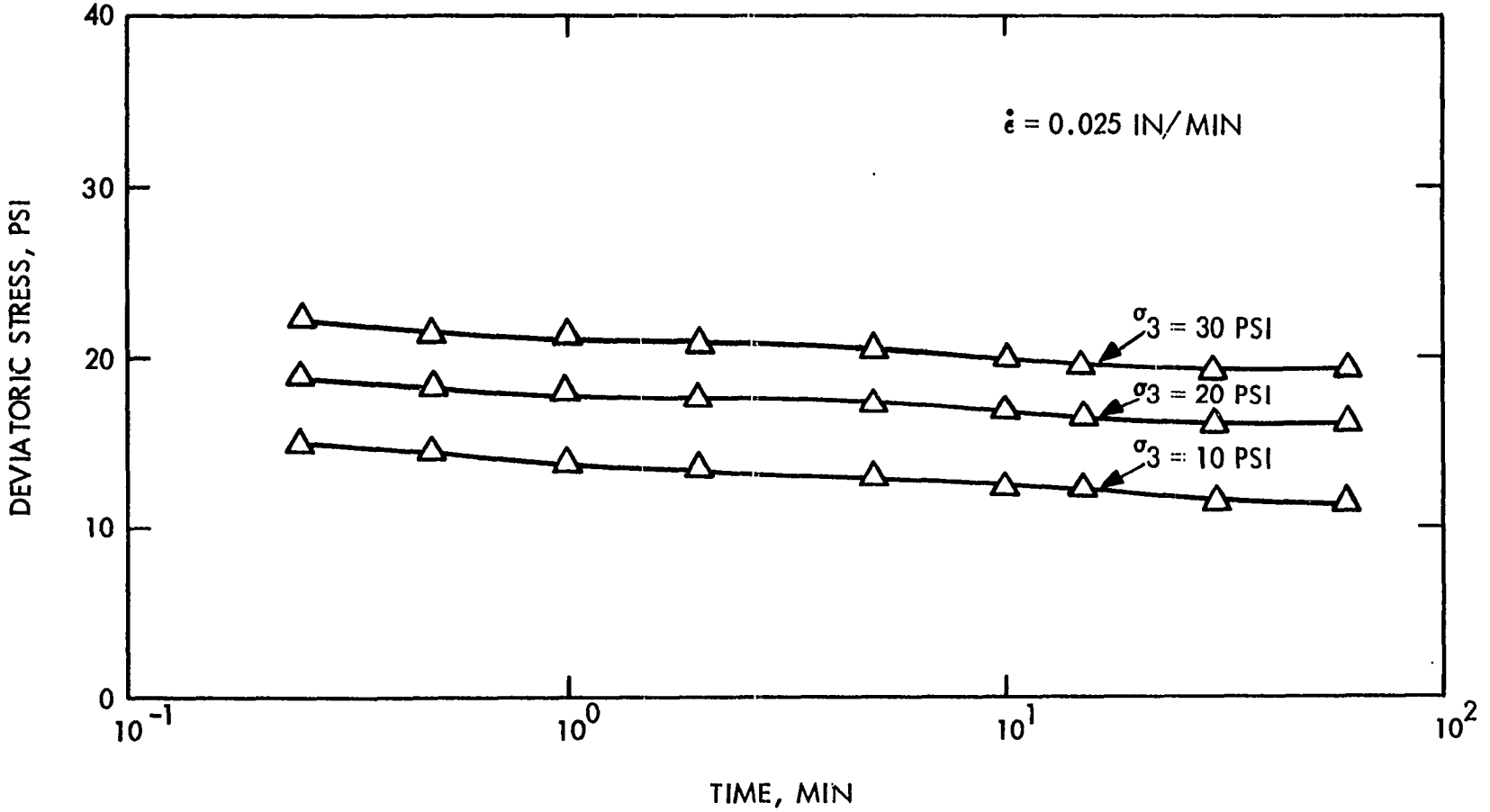


Figure 5.23 Effect of lateral pressure on relaxation characteristics of ultrasonic treated shale 15.

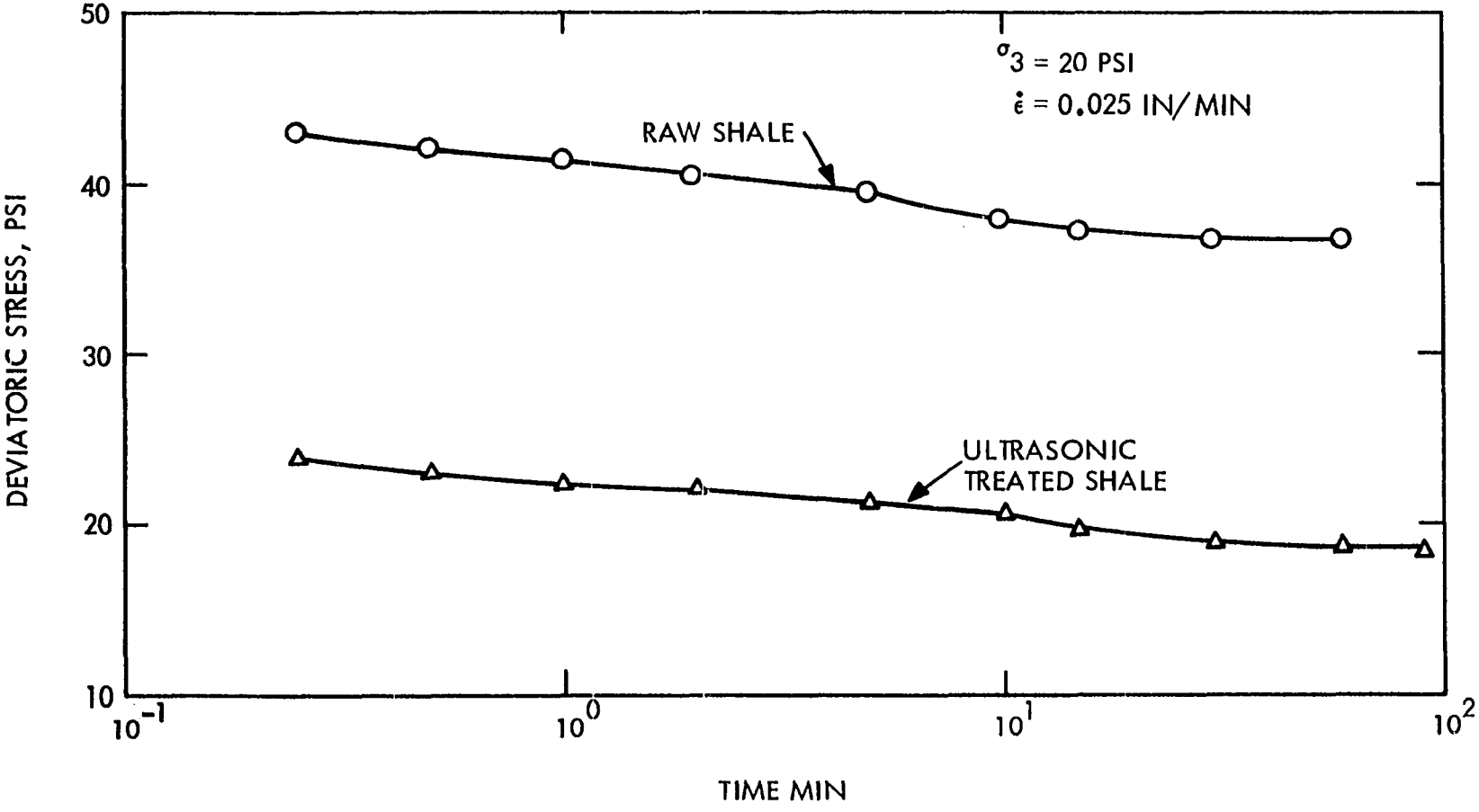


Figure 5.24 Effect of ultrasonic treatment on relaxation characteristics of shale 24.

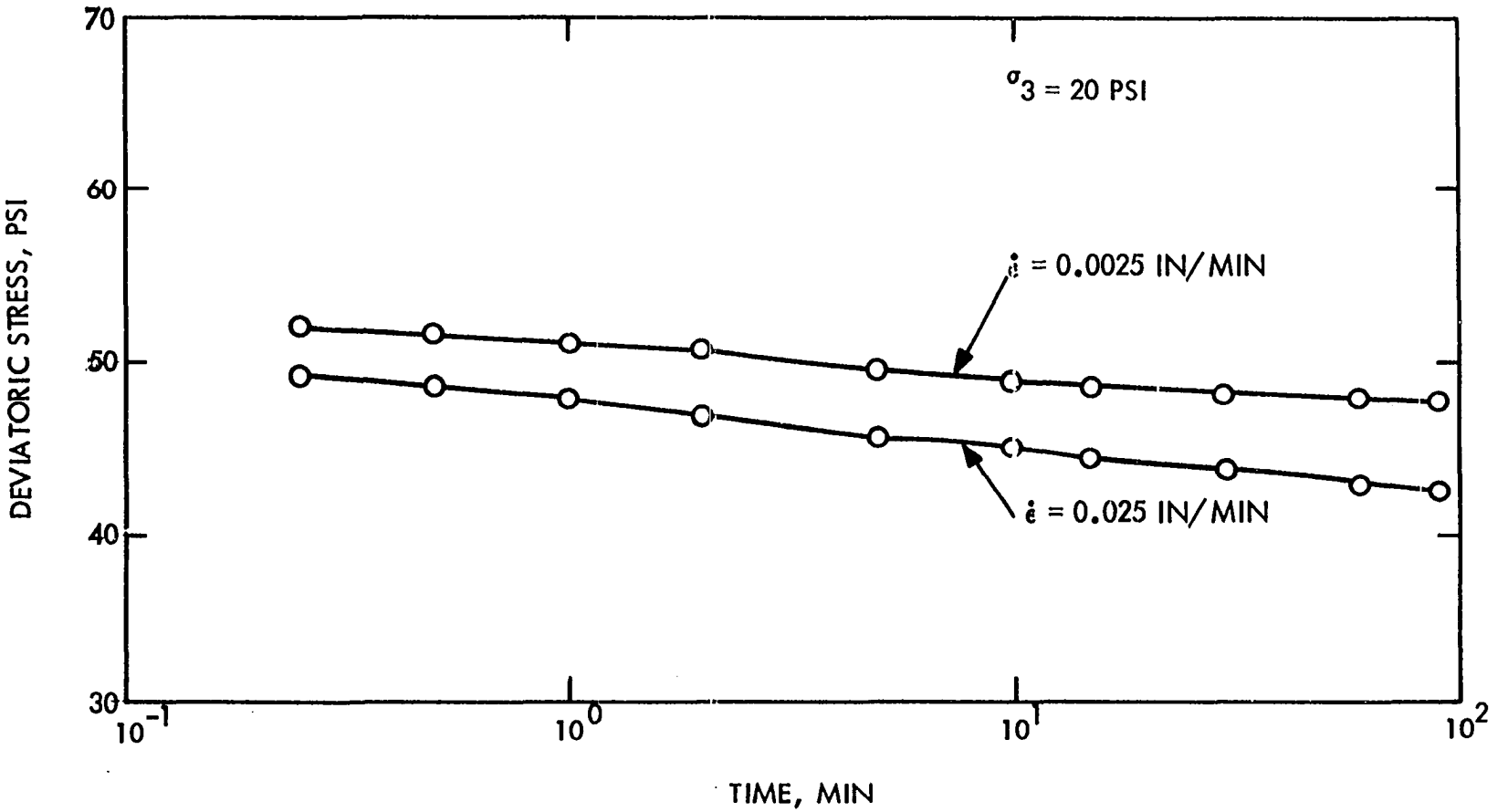


Figure 5:25 Effect of strain rate on relaxation characteristics of raw shale 24.

ultrasonic treated shales were lower than those for raw shales. Only exception was shale 13 for which raw shales had less strength, as seen from the Table 5.7.

The values of strength parameters were calculated corresponding to the values of maximum and minimum principal stresses at the beginning and end of relaxation and presented in Table 5.8. Cohesion decreased during relaxation, the decreases ranging from 0.2 psi to 4.0 psi irrespective of either treatment or strain rate. However, angle of friction showed both increase and decrease, ranging from -1.4 degree to +2.9 degree. Variations of these could be due to the dissipation of pore water pressure during relaxation and movement of free water in the pores.

Figure 5.25 shows the effect of strain rate on relaxation characteristics. Both raw and ultrasonic treated samples for all shales were affected in a similar manner. The decrease of deviatoric stress during relaxation expressed as a percentage of the deviatoric stress before relaxation is termed as percent relaxation of deviatoric stress and the average values of percent relaxation were plotted in Figure 5.26. The values of percent relaxation for strain rate of 0.025 in/min reduced approximately by 50 percent at low strain rate of 0.0025 in/min.

At low strain rate, consolidation of the specimen under both normal and lateral pressures took place for a

TABLE 5.8

STRENGTH PARAMETERS AT THE BEGINNING AND END OF RELAXATION

Shale Number*	Speed in/min	Cohesion psi		Δ Cohesion	Angle of Friction degrees		Δ Friction
		Begin	End		Begin	End	
13R	0.025	9.8	5.8	-4.0	11.9	14.8	+2.9
13U	0.025	14.0	11.7	-2.3	9.8	8.7	-1.1
13R	0.0025	14.4	13.5	-0.9	7.2	6.9	-0.3
13U	0.0025	14.4	12.9	-1.5	9.1	9.0	-0.1
15R	0.025	13.4	9.5	-3.9	11.9	12.2	+0.3
15U	0.025	5.6	3.3	-2.3	8.5	9.4	+0.9
15R	0.0025	8.0	7.8	-0.2	13.0	11.8	-1.2
15U	0.0025	7.7	7.0	-0.7	4.6	4.8	+0.2
21R	0.025	16.2	12.9	-3.3	9.4	8.0	-1.4
21U	0.025	7.6	3.9	-3.7	9.8	11.3	+1.5
21R	0.0025	17.2	15.4	-1.8	13.1	12.5	-0.6
21U	0.0025	12.8	12.0	-0.8	4.0	3.6	-0.4
24R	0.025	16.4	13.3	-3.1	12.1	11.5	-0.6
24U	0.025	14.1	10.6	-3.5	10.3	10.9	+0.6
24R	0.0025	17.1	14.9	-2.2	11.1	11.9	+0.8
24U	0.0025	10.2	9.7	-0.5	11.1	10.3	-0.8

*R - raw shale and U - ultrasonic treated shale.

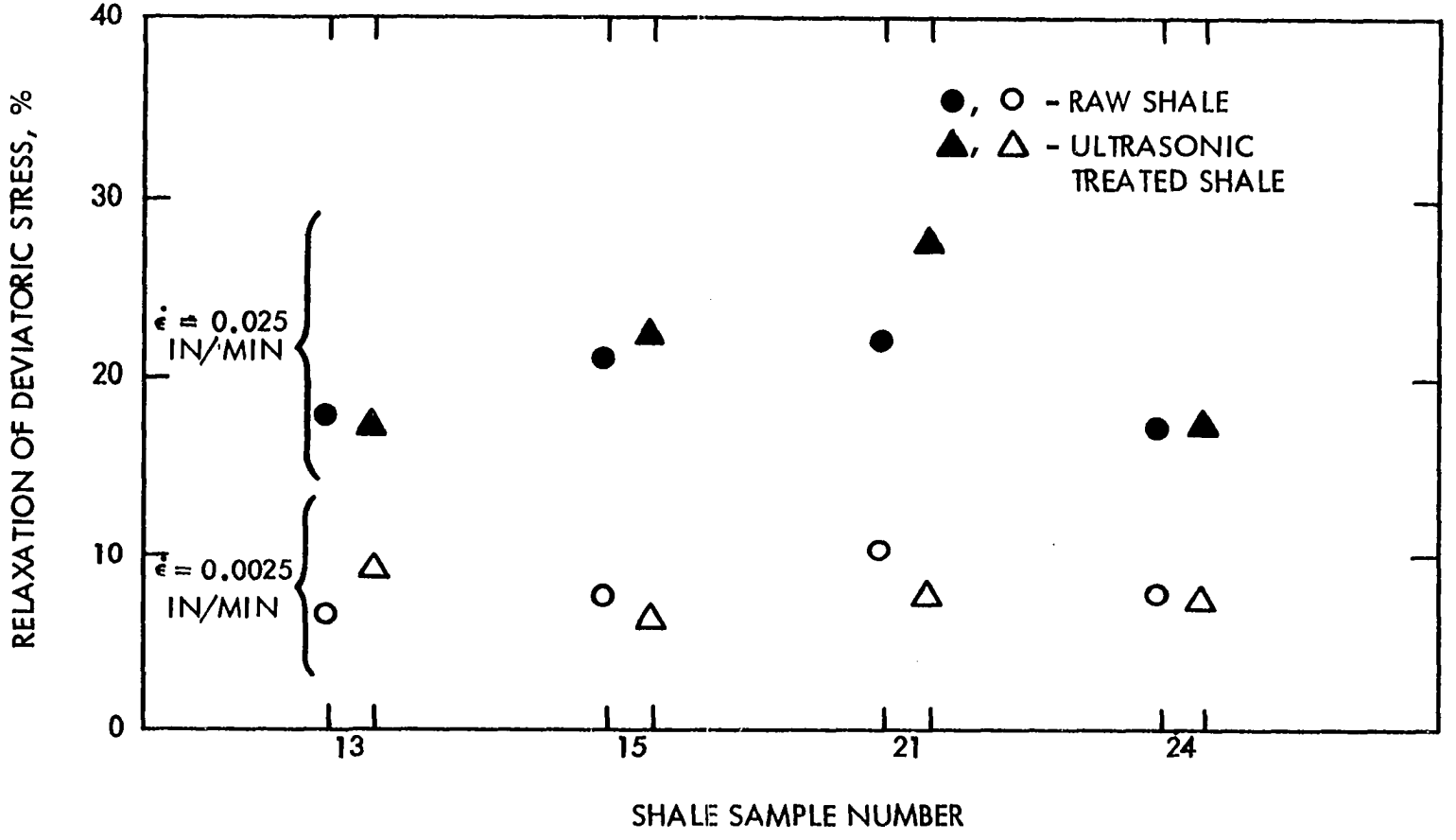


Figure 5.26 Effect of strain rate on percent relaxation of deviatoric stress for all shales.

longer time. Also there were possibly some loss of water and thixotropic stiffening of the soil particles. Consequently the percent relaxation of deviatoric stress was reduced.

The relaxation characteristics are usually dependent on the strain level, stress, water content and strain rate. Ultrasonic treatment did not indicate any influence on the relaxation properties as average value of percent relaxation remained more or less the same after treatment. However, among the variables studied, strain rate seemed to have more influence than others.

Volume Change Test Results

The data pertaining to volume change are given in Table 5.9 and graphical representations are plotted in Figures 5.27 through 5.30. Shale 13 with montmorillonite as the predominant clay mineral and shale 21 which contains montmorillonite illite mixed layer clay minerals exhibited a pattern different from that of shales 15 and 24 which are predominantly illite. For shales 15 and 24, considerable increases in volume change were noticed as a consequence of ultrasonic treatment (from 0.80 percent to 6.44 percent for shale 15 and from 9.05 percent to 18.77 percent for shale 24). The amount less than 2 micron clay and clay size particles increased greatly after ultrasonic treatment and resulted in large increases in the surface area. The increase in volume change is primarily due to the increased surface

TABLE 5.9

VOLUME CHANGE TEST DATA

Shale Number	Volume Change %	Test Time hr	Increase in Moisture Content* %
Raw			
13	10.30	152	47.4
15	0.80	48	20.7
21	8.95	144	50.6
24	9.05	180	50.2
Ultrasonic			
13	12.41	239	60.8
15	6.44	104	31.5
21	11.60	300	48.7
24	18.77	157	87.7

*Percent increase over the molding water content.

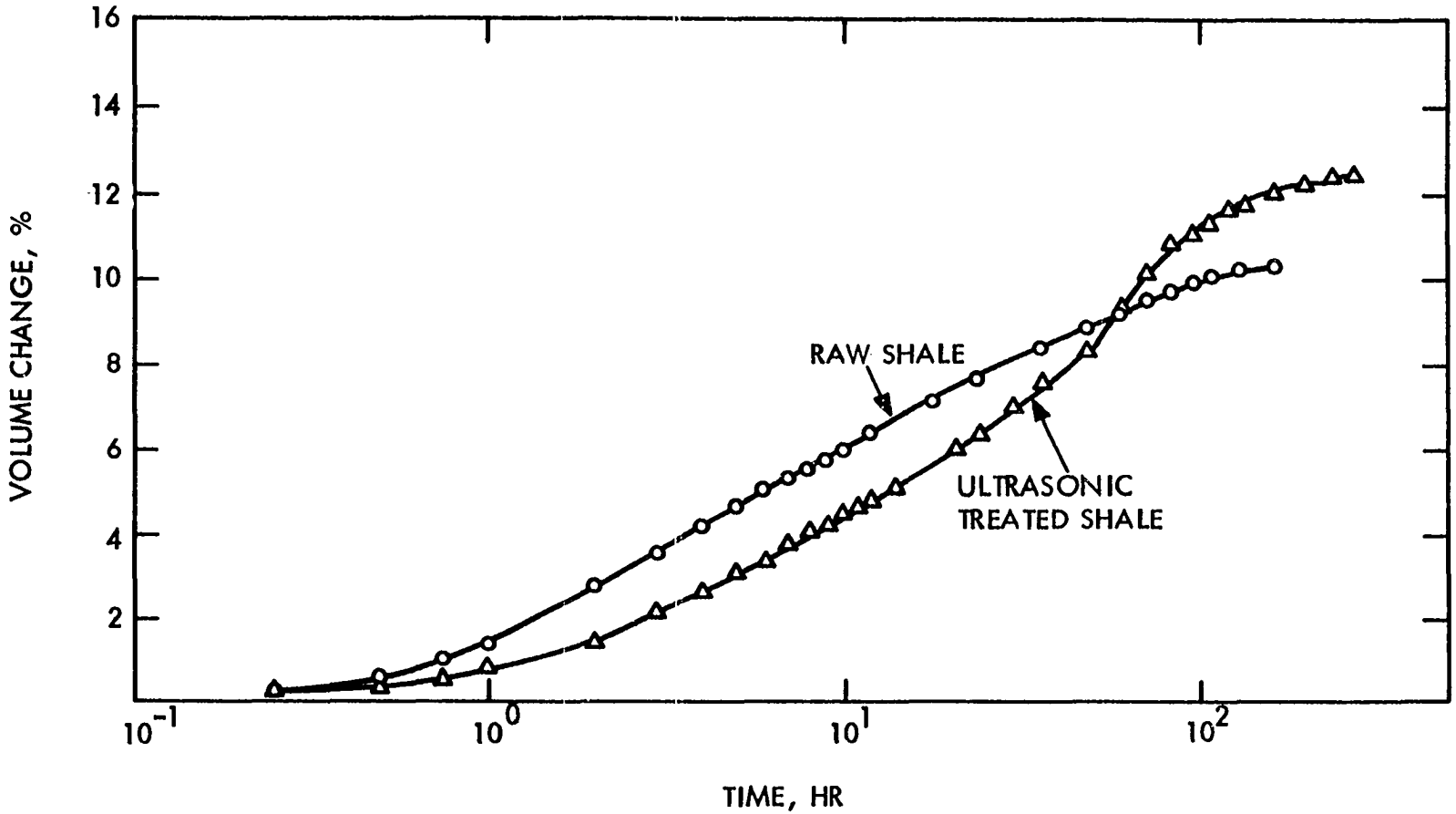


Figure 5.27 Effect of ultrasonic treatment on time dependency of volume change for shale 13.

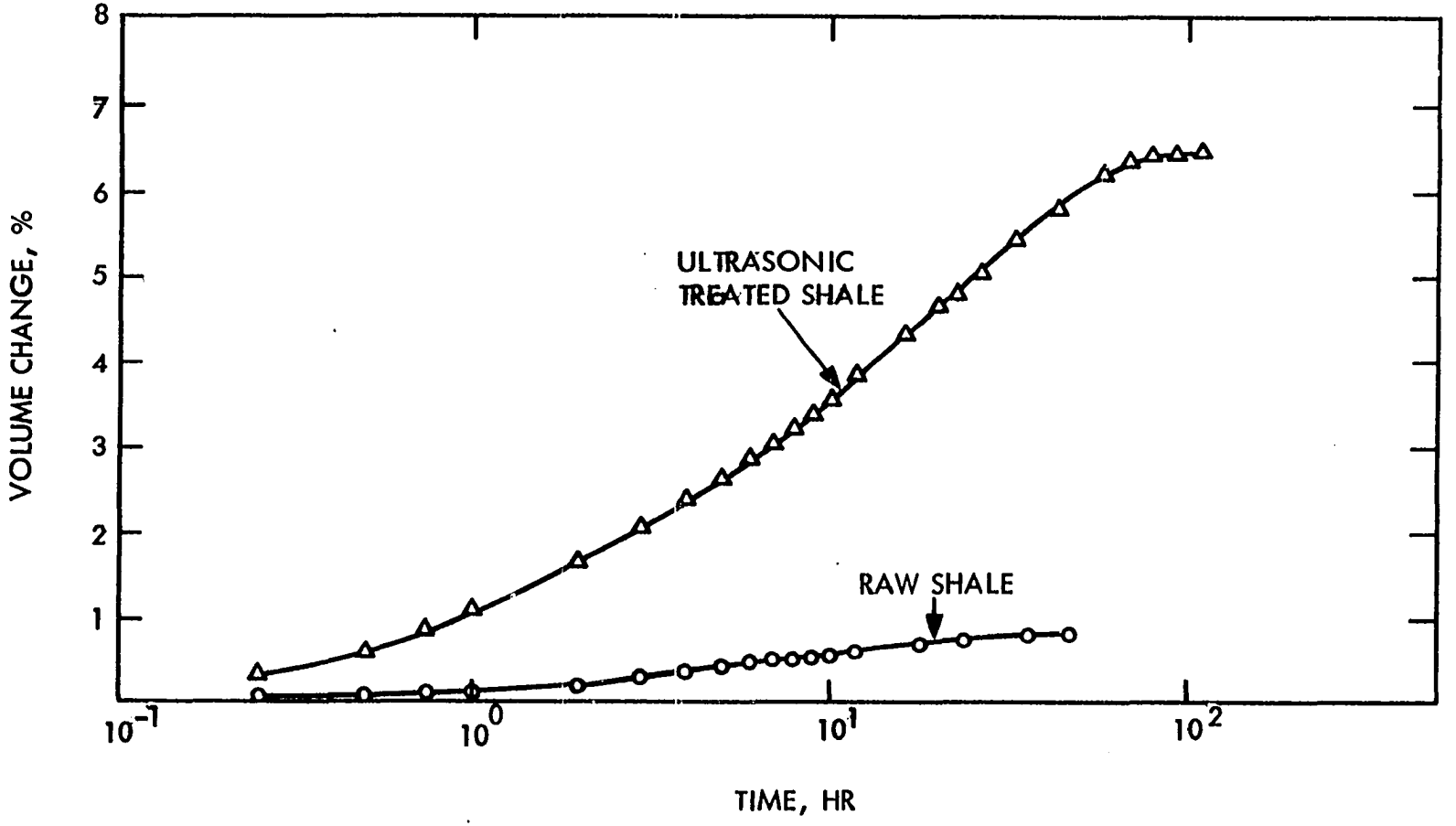


Figure 5.28 Effect of ultrasonic treatment on time dependency of volume change for shale 15.

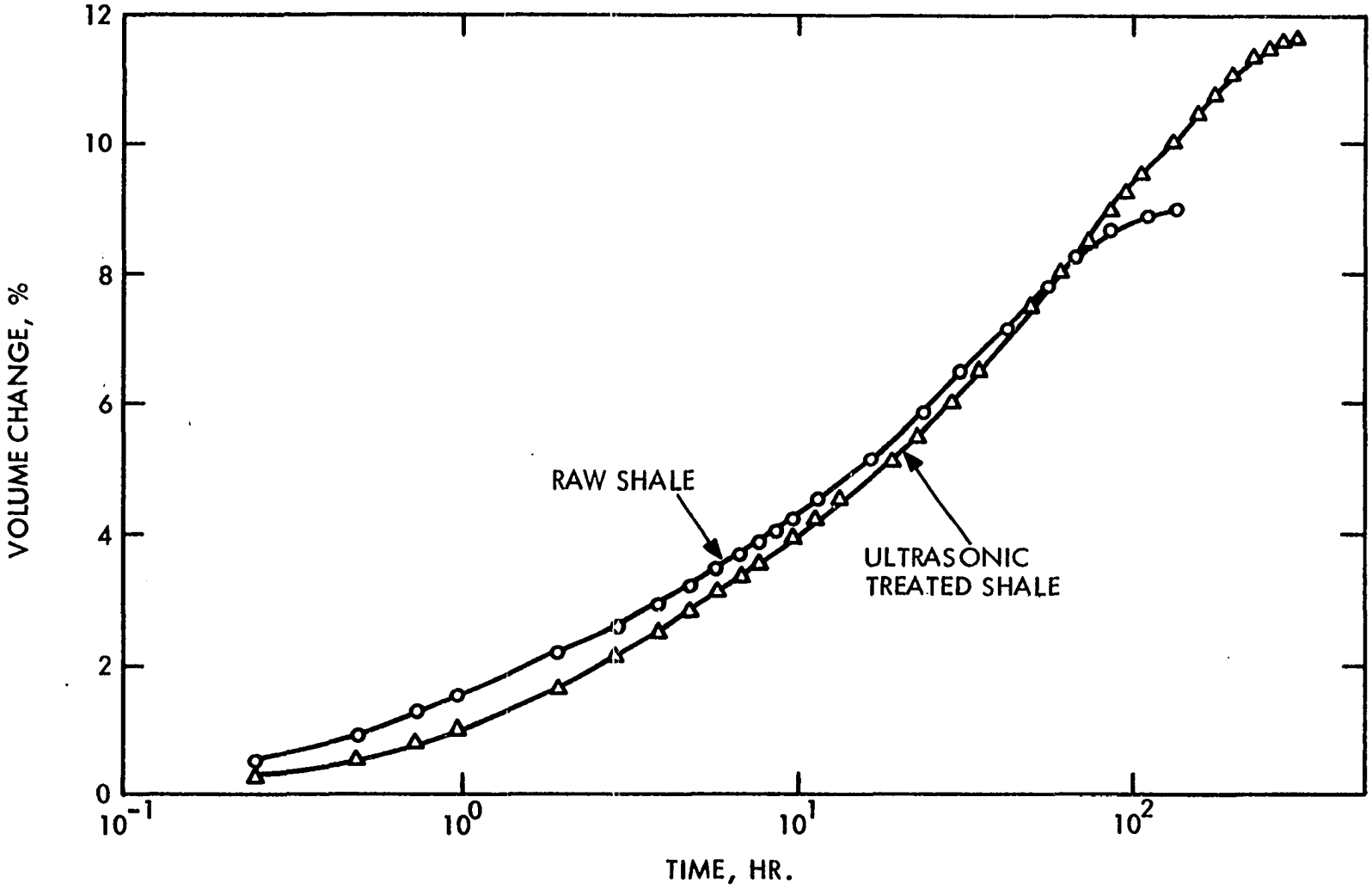


Figure 5.29 Effect of ultrasonic treatment on time dependency of volume change for shale 21.

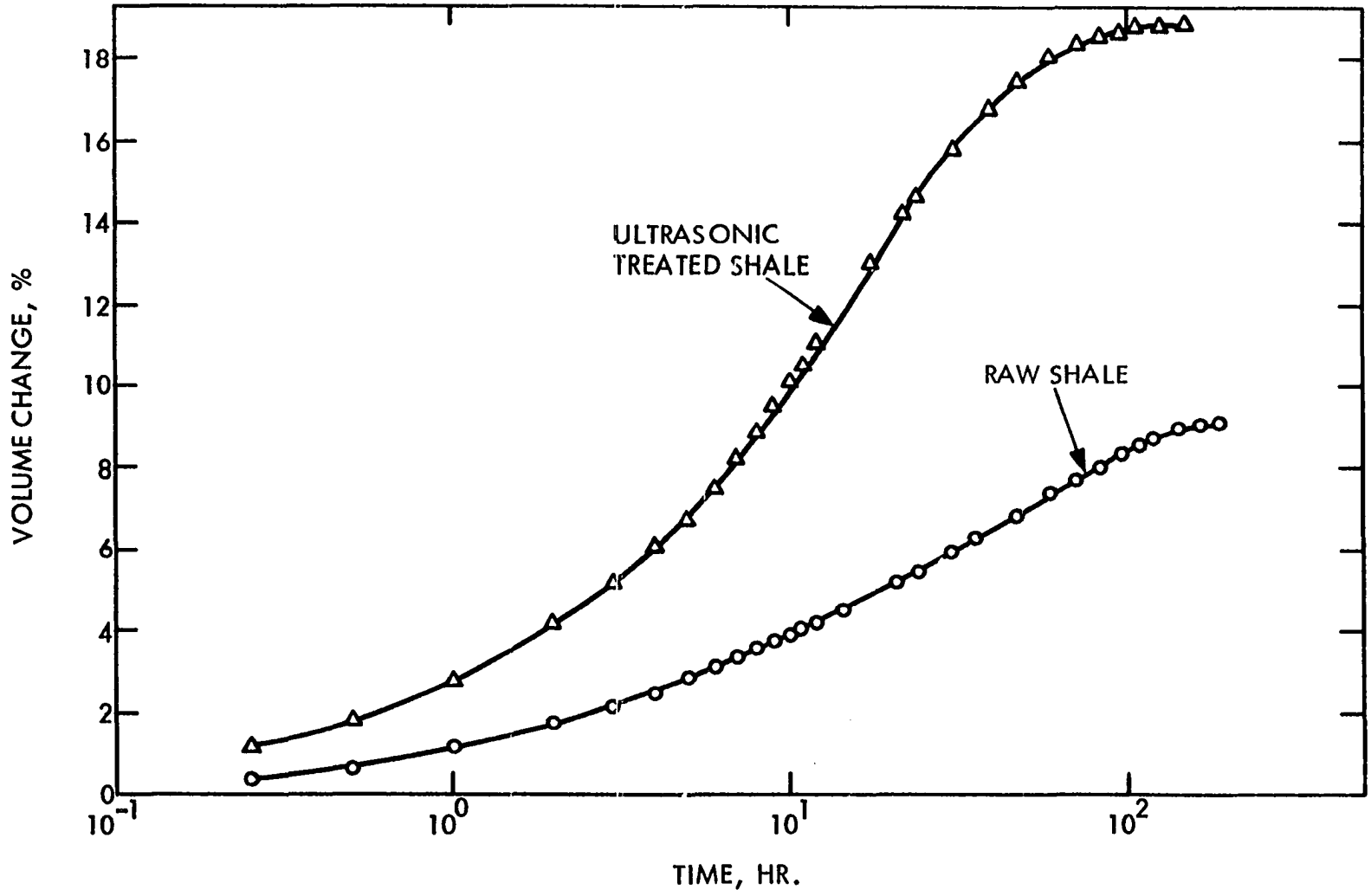


Figure 5.30 Effect of ultrasonic treatment on time depeneency of volume change for shale 24.

hydration occurring in the clay and clay size particles. This phenomenon is very typical of illitic clay minerals for which the volume change characteristics are controlled by surface hydration of clay surfaces.

The other mechanism controlling the volume change is the double layer expansion which is predominant in montmorillonitic clay minerals. Probably the volume change characteristics, of shales 13 and 21, were controlled primarily by the latter mechanism as shale 13 and 21 had more montmorillonite besides other clay minerals. Unlike shales 15 and 24, shales 13 and 21 showed only small increases in volume change after ultrasonic treatment (from 10.3 percent to 12.4 percent for shale 13 and 8.95 percent to 11.6 percent for shale 21).

The volume change - time relationships were also different. For the first 60 to 70 hours of the test, volume change of ultrasonic treated shale was lower than that of raw shale. However, after this period, the volume change of ultrasonic treated shale showed small increase over that of raw shale. Also the total time for the test increased significantly for shales 13 and 21 after ultrasonic treatment (from 152 hrs to 239 hrs for shale 13 and from 144 hrs to 300 hrs for shale 21). That the volume change - time relation ships for shales 15 and 24 were different from those for shales 13 and 21, as a consequence of ultrasonic treatment, might imply that the mechanisms characteristic of the

predominant clay minerals in the shales were influenced differently by ultrasonic treatment.

In general, the increases in volume change for all shales seemed to be proportional to the increase in the amount of clay and clay size particles.

The rate and magnitude of volume change would depend upon the chemical and mineralogical characteristics of clay particles, amount of clay content, initial density and moisture content. Supported by experimental evidence, it is generally assumed that the chemical and mineralogical properties of clay are adequately represented by Atterberg limits. Density and molding water content are also important as the initial placement of the soil decides the "structure."

Considering the data pertaining to all 24 shale samples from the referenced study (Laguros, 1972), a statistical analysis was run. A new parameter, termed as "reaction potential" was developed to quantify both type and amount of clay minerals. The amount of 2 micron clay and clay size particles and the percentages of different clay minerals present are required for calculation of reaction potential. For example, shale 13 had 11 percent kaolinite and 89 percent montmorillonite as clay minerals and 52 percent less than 2 micron size particles (Table 5.10). Assuming the average value of cation exchange capacity for kaolinite as 10 meq/100 gm and 115 meq/100 gm for montmorillonite, the total reaction

TABLE 5.10

CALCULATIONS OF REACTION POTENTIAL

Shale No.	Type of Clay Mineral, %				Total Reaction Potential[c]				SUM	% 2μ	RP[d]
	I	K	M	ML[a]	I (25)	K (10)	M (115)	ML (70)[b]			
6	2	3	20	75	0.5	0.3	23.0	52.5	76.3	57	43.5
7	78	13	13	--	19.5	1.3	14.95	--	35.8	8	2.9
8	100	--	--	--	25	--	--	--	25.0	2	0.5
9	59	25	16	--	14.75	2.5	18.40	--	35.7	10	3.6
10	60	21	19	--	15	2.1	21.85	--	39.0	39	15.2
11	23	7	70	--	5.75	0.7	80.50	--	87.0	31	27.0
11A	17	5	29	49	4.25	0.5	33.4	34.3	72.5	29	21.0
12	16	6	56	22	4.0	0.6	64.4	15.4	84.4	70	59.1
13	--	11	89	--	--	1.1	102.35	--	103.5	52	53.8
14	69	24	7	--	17.25	2.4	8.05	--	27.7	8	2.2
15	58	24	18	--	14.50	2.4	20.7	--	37.6	9	3.4
16	80	5	15	--	20.0	0.5	17.25	--	37.8	10	3.8
17	80	10	10	--	20.0	1.0	11.50	--	32.5	19	6.2
18	91	5	4	--	22.75	0.5	4.6	--	27.9	58	16.2
19	82	10	8	--	20.50	1.0	9.2	--	30.7	29	8.9
20	43	11	19	27	10.75	1.1	21.85	18.9	52.6	39	20.5
21	15	6	22	57	3.75	0.6	25.30	39.9	69.6	28	19.7
22	19	26	21	34	4.75	2.6	24.15	23.8	55.3	63	34.8
23	43	16	19	22	10.75	1.6	21.85	15.4	49.6	34	16.9
24	68	28	4	--	17.0	2.8	4.6	--	24.4	23	5.6
25	59	28	--	13	14.75	2.8	--	9.1	26.7	9	2.4
26	15	79	--	6	3.75	7.9	--	4.2	15.9	54	8.6
27	53	--	--	47	13.25	--	--	32.9	46.2	13	6.0
28	100	--	--	--	25	--	--	--	25.0	6	1.5

[a] I = Illite; K = Kaolinite; M = Montmorillonite; ML = Mixed layer, Illite-Montmorillonite.

[b] Average cation exchange capacity of clay minerals (Grim, R. E., (1968). Clay Mineralogy, McGraw-Hill, New York, p. 189).

[c] (Average CEC) (Proportion of type of clay mineral).

[d] (2μ clay) (Total reaction potential)/100.

potential for 100 gm of clay content would be calculated as

$$(11/100)(10) + (89/100)(115) = 103.5 \text{ meq/100 gm}$$

Since shale 13 has only 52 percent clay content the reaction potential would be

$$(52/100)(103.5) = 53.8 \text{ meq/100 gm of shale}$$

The calculations for all shales are shown in Table 5.10. The data on volume change, moisture-density, clay content, liquid limit, plasticity index and reaction potential are presented in Table 5.11. Since the reaction potential is a composite parameter calculated from both type and amount of clay minerals, it is considered to be better than liquid limit and plasticity index.

The dependent variables considered are volume change and logarithm of volume change. The values of correlation coefficients between the dependent variables and moisture content, density, liquid limit, plasticity index, clay content and reaction potential are presented in Table 5.12 and 5.13.

As evident from the Table 5.12, the single predictive parameter seemed to be the reaction potential. The plasticity index and the amount of particles less than 2 micron size were also good predictive parameters. However, the reaction potential would be preferred as it would indicate both

TABLE 5.11

VALUES OF VOLUME CHANGE, DENSITY, MOISTURE CONTENT, INDEX PROPERTIES AND REACTION POTENTIAL FOR RAW SHALES

Shale Number	Volume Change %	Dry Density pcf	Optimum Moisture Content %	Amount Less Than 2 Micron %	Liquid Limit %	Plasticity Index %	Reaction Potential meq/100 gm
6	13.65	93.6	28.5	57	36	12	43.5
7	5.67	116.7	14.7	8	28	2	2.9
8	0.64	110.8	13.8	2	0	0	0.5
9	3.78	122.0	13.0	10	26	5	3.6
10	9.32	108.0	19.2	39	46	23	15.2
11	5.67	107.6	18.2	31	44	14	27.0
11A	4.12	103.5	22.0	29	40	8	21.0
12	18.10	90.1	28.0	70	83	38	59.1
13	10.30	110.6	18.4	52	47	26	53.8
14	1.85	118.3	14.5	8	26	7	2.2
15	0.80	119.5	13.5	9	33	13	3.4
16	1.85	105.7	21.4	10	38	14	3.8
17	1.35	107.6	20.4	19	31	7	6.2
18	3.30	91.8	28.5	58	41	11	16.2
19	3.57	100.1	23.4	29	39	14	8.9
20	9.26	109.5	18.7	39	40	17	20.5
21	8.95	109.0	19.5	28	29	8	19.5
22	15.15	92.8	27.0	63	64	29	34.8
23	6.35	107.2	19.5	34	37	12	16.9
24	9.05	112.2	17.3	23	38	13	5.6
25	7.05	112.5	14.3	9	30	8	2.4
26	7.62	102.5	19.5	54	36	13	8.6
27	4.83	95.2	24.5	13	34	5	6.0
28	0.26	103.3	19.7	6	0	0	1.5

TABLE 5.12

PROPERTY INTERRELATIONSHIPS BETWEEN VOLUME CHANGE AND INDEX PROPERTIES, DENSITY, OPTIMUM MOISTURE CONTENT AND REACTION POTENTIAL FOR OKLAHOMA SHALES

Variable	Dry Density	Optimum Moisture Content	Liquid Limit	Plasti-city Index	2 micron Clay	Reaction Potential	Intercept	r^a	SEE^b
V	-0.489	0.481	0.762	0.790	0.789	0.799	--	--	--
V	--	--	--	--	--	0.231	2.735	0.799	2.91
V	--	--	--	--	0.182	--	1.017	0.789	2.89
V	--	--	--	0.406	--	--	1.243	0.790	2.88
V	--	--	0.225	--	--	--	-1.804	0.762	3.04
V	-0.116	-0.185	--	--	--	0.230	18.720	0.802	3.03
V	-0.141	-0.407	--	--	0.205	--	23.502	0.802	2.92
V	-0.105	-0.029	--	0.367	--	--	13.544	0.805	2.90
V	-0.241	-0.373	0.217	--	--	--	31.626	0.783	3.04

r^a = correlation coefficient.

SEE^b = standard error of estimate.

TABLE 5.13

PROPERTY INTERRELATIONSHIPS BETWEEN LOGARITHM OF VOLUME CHANGE AND INDEX PROPERTIES, DENSITY, OPTIMUM MOISTURE CONTENT AND REACTION POTENTIAL FOR OKLAHOMA SHALES

Variable	Dry Density	Optimum Moisture Content	Liquid Limit	Plasti-city Index	2 micron Clay	Reaction Potential	Intercept	r[a]	SEE[b]
Log V	-0.409	0.356	0.702	0.645	0.654	0.630	--	--	--
Log V	--	--	--	--	--	0.018	0.355	0.630	0.37
Log V	--	--	--	--	0.017	--	0.125	0.654	0.39
Log V	--	--	--	0.037	--	--	0.153	0.645	0.40
Log V	--	--	0.023	--	--	--	-0.218	0.702	0.37
Log V	0.019	0.034	--	--	--	0.017	-2.302	0.638	0.38
Log V	-0.031	-0.080	--	--	0.020	--	4.925	0.700	0.39
Log V	-0.028	-0.041	--	0.034	--	--	3.977	0.673	0.40
Log V	-0.042	-0.086	0.025	--	--	--	5.923	0.754	0.36

a - correlation coefficient.

b - standard error of estimate.

the amount of clay size particles and the nature of clay minerals. For the variable logarithm of volume change, the liquid limit was the best single predictive parameter (Table 5.13).

The regression equation between volume change and reaction potential was

$$V = 2.735 + 0.231\ (RP) \qquad (5.8)$$

where V = volume change, percent

RP = reaction potential, meq/100 gm

The correlation coefficient was 0.799 and standard error of estimate was 2.91.

The regression equation between volume change and plasticity index was

$$V = 1.243 + 0.406\ (PI) \qquad (5.9)$$

where V = volume change, percent

PI = plasticity index, percent

The correlation coefficient was 0.79 and standard error of estimate was 2.88.

For the variable, logarithm of volume change and liquid limit, the best line of fit was obtained as

$$\log V = -0.218 + 0.023\ (LL) \qquad (5.10)$$

where V = volume change, percent

LL = liquid limit, percent

The correlation coefficient was 0.70 and standard error of estimate was 0.37.

If placement conditions were considered, then the best multiple regression line for the variable volume change was found to be

$$V = 18.72 - 0.116\ (DD) - 0.185\ (OMC) + 0.230\ (RP) \tag{5.11}$$

where V = volume change, percent

DD = dry density, pcf

OMC = optimum moisture content, percent

RP = reaction potential, meq/100 gm

The correlation coefficient was 0.802 and standard error of estimate was 3.03.

With plasticity index as the variable, the equation was

$$V = 13.544 - 0.105\ (DD) - 0.029\ (OMC) + 0.367\ (PI) \tag{5.12}$$

where V = volume change, percent

DD = dry density, pcf

OMC = optimum moisture content, percent

PI = plasticity index, percent

The multiple correlation coefficient was 0.805 and standard error of estimate was 2.90. With logarithm of volume change

as the dependent variable, the best multiple regression line was obtained as

$$\log V = 5.923 - 0.042(DD) - 0.086(OMC) + 0.025(LL) \quad (5.13)$$

where V = volume change, percent

DD = dry density, pcf

OMC = optimum moisture content, percent

LL = liquid limit, percent.

The multiple correlation coefficient was 0.754 and standard error of estimate was 0.357.

The values of volume change for ultrasonic treated shale samples were calculated by those six equations and compared with the actual values in Table 5.14.

Although the correlation coefficients seemed to be higher for the semi-logarithmic relationships than for normal relationships, the values predicted by semi-logarithmic relationships spread over a very wide range. The values predicted by normal relationships were closer to the actual values.

If type and amount of clay minerals are known then the Equations 5.8 and 5.11 involving the reaction potential could be used. Otherwise Equations 5.9 and 5.12 involving the plasticity index could be used for prediction. However, the Equations 5.8, 5.9, 5.11 and 5.12 did not predict the volume change for shale 24. The upper limit values given by the logarithmic relationships involving the liquid limit were closer to the actual values for this shale.

TABLE 5.14

COMPARISON OF THE PREDICTED VALUES OF VOLUME CHANGE OF ULTRASONIC TREATED SHALES WITH THEIR ACTUAL VALUES

	Method	Shale Number			
		13	15	21	24
1.	Actual value	12.41	6.44	11.60	18.77
2.	By Equation 5.8	13.69 - 19.51	5.04 - 10.86	7.38 - 13.20	2.93 - 8.75
3.	By Equation 5.9	10.12 - 15.88	6.45 - 12.21	3.61 - 9.37	6.45 - 12.21
4.	By Equation 5.10	4.57 - 23.97	2.48 - 13.00	1.34 - 7.03	3.14 - 16.45
5.	By Equation 5.11	13.43 - 19.49	4.55 - 10.61	7.21 - 13.27	2.77 - 8.83
6.	By Equation 5.12	10.48 - 16.28	7.15 - 12.95	5.77 - 11.57	8.09 - 13.89
7.	By Equation 5.13	3.85 - 21.14	2.80 - 15.38	1.48 - 8.15	3.12 - 17.10

Consolidation Test Results

The results of consolidation tests on both raw and ultrasonic treated shales are presented in Table 5.15. The e-log p relationships are shown in Figures 5.31 through 5.34. The e-log p curves, after treatment, became steeper. The amount of decompression was also more for the treated shale. Bjerrum (1967) reported that weathering destroyed the bonds and liberated the locked-in recoverable strain energy causing the clay to expand, its water content to increase and its shear strength to decrease. In the case of remolded clays, large increases in swelling on removal of soil pressure were observed. This was due to the disintegration of diagenetic bonds in the process of remolding (Figure 5.35).

In this study, all shale materials used were disturbed and remolded. The ultrasonic treatment causes further breakdown of the bonds and disaggregation of the particles. Effect of the destruction of bonds by ultrasonic treatment caused the decompression to increase during rebound.

Figures 5.36 and 5.37 show the relationship between consolidation stress and axial strain for shales 13 and 24, respectively. This is very typical of one-dimensional confined compression tests. The strain increased non-linearly with the stress. The stress-strain curve became approximately parallel to the axis of stress. Shales 15 and 21 exhibited the same pattern as shale 24. Among all shales shale 13 was the least affected by ultrasonic treatment. The optimum

TABLE 5.15

CONSOLIDATION TEST DATA

Shale Number	Compression Void Ratio	Decompression Void Ratio	DCR	Coefficient of Consolidation 10^{-3} cm^2/sec	Coefficient of Permeability 10^{-8} cm/sec
Raw					
13	0.168	0.066	0.3660	5.97	4.84
15	0.116	0.028	0.2440	6.52	3.91
21	0.177	0.073	0.4150	4.60	2.81
24	0.190	0.078	0.4145	2.37	2.08
Ultrasonic					
13	0.160	0.071	0.4445	2.40	2.35
15	0.180	0.065	0.3605	1.90	1.05
21	0.279	0.101	0.3615	2.20	1.66
24	0.292	0.129	0.4425	2.15	1.80

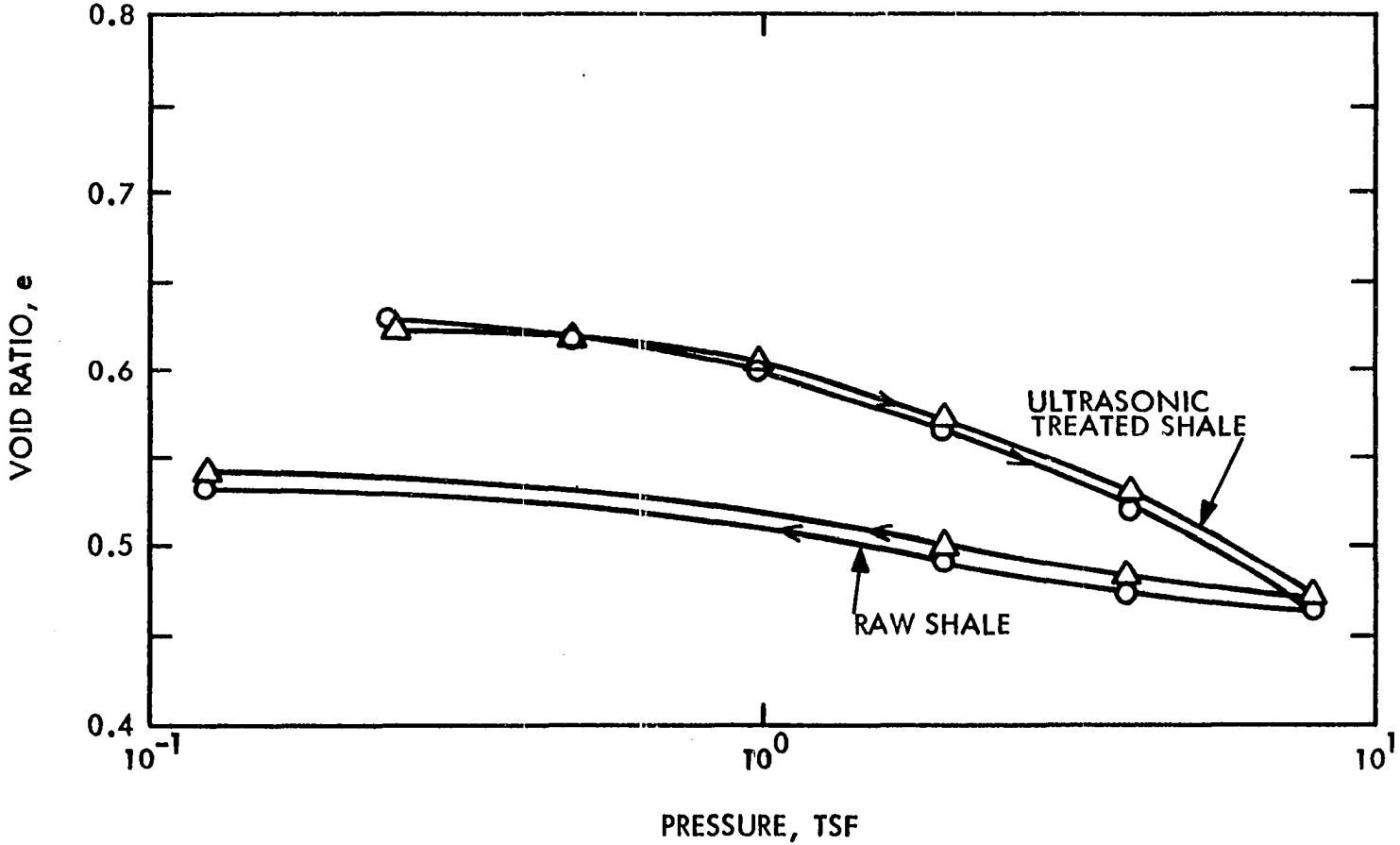


Figure 5.31 Effect of ultrasonic treatment on e-log p relationship for shale 13.

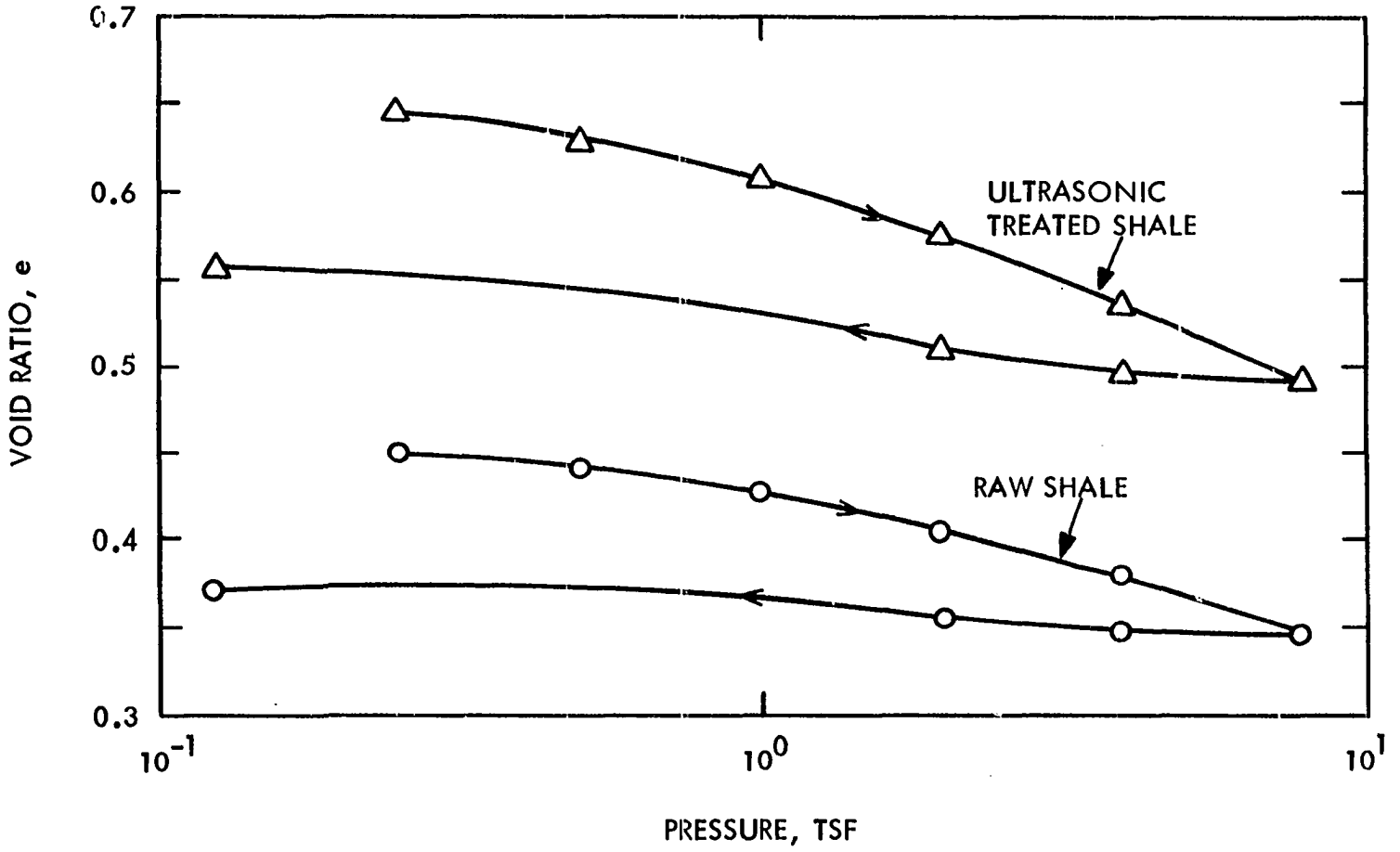


Figure 5.32 Effect of ultrasonic treatment on e-log p relationship for shale 15.

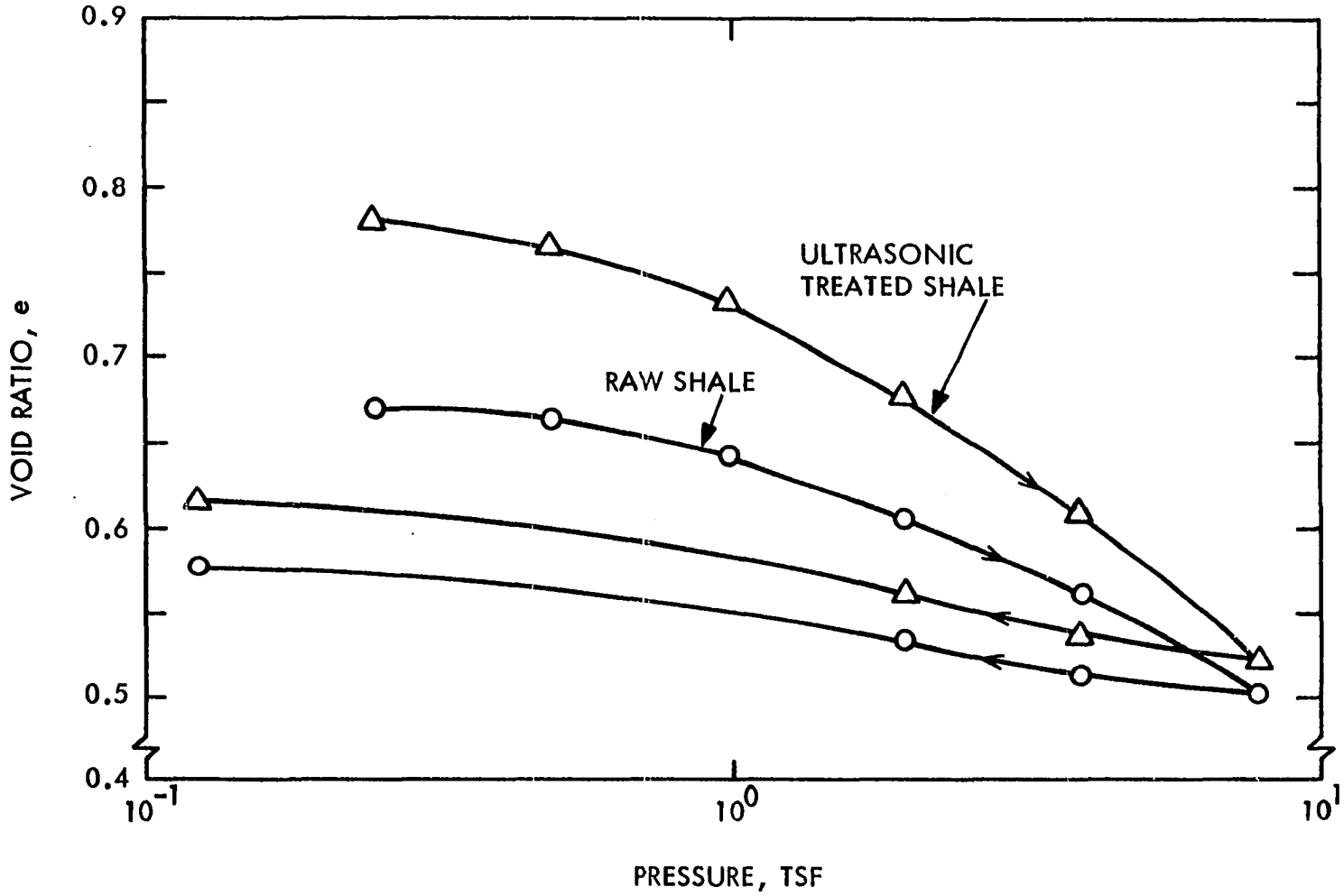


Figure 5.33 Effect of ultrasonic treatment on e-log p relationship for shale 21.

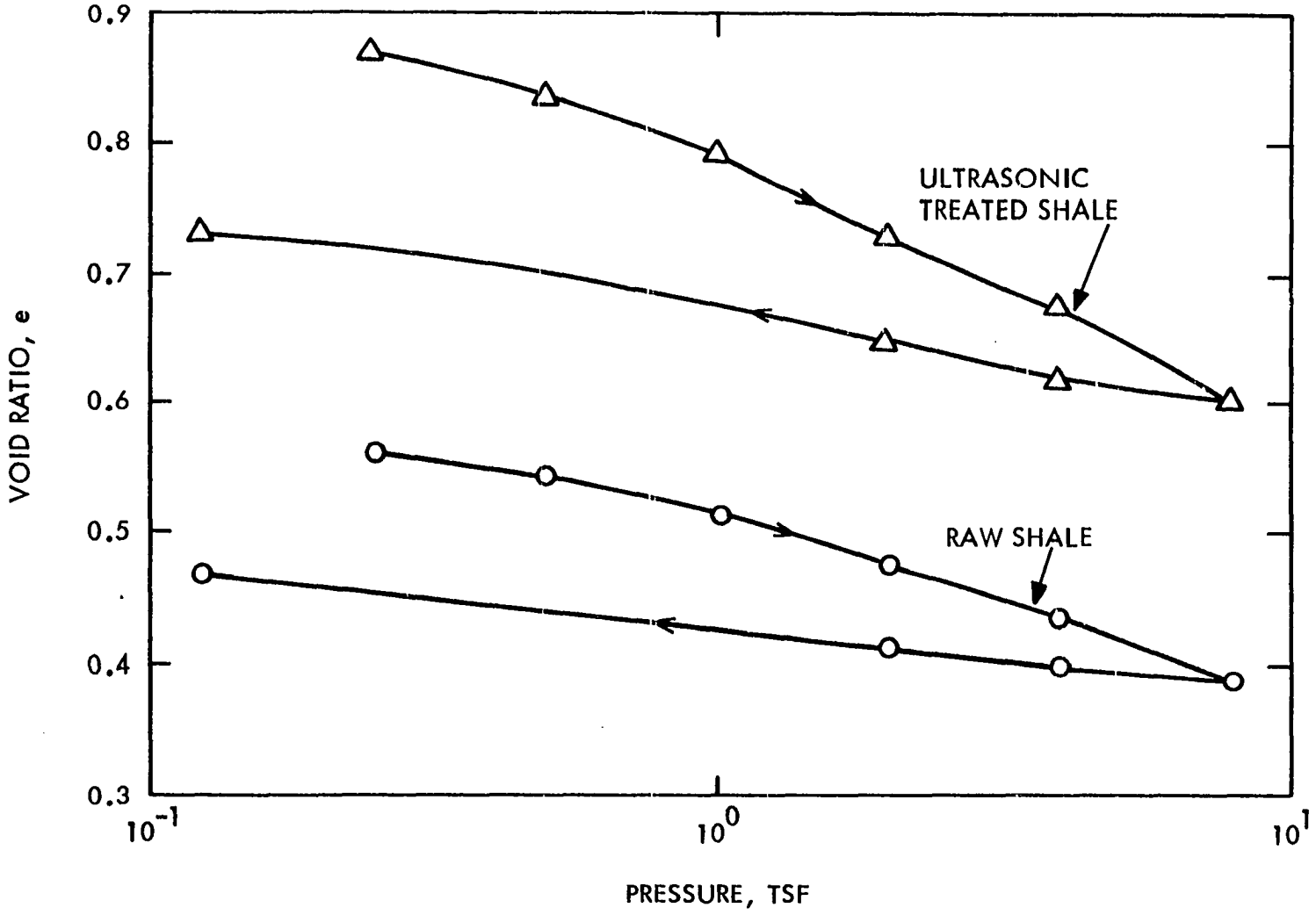


Figure 5.34 Effect of ultrasonic treatment on e-long p relationship for shale 24.

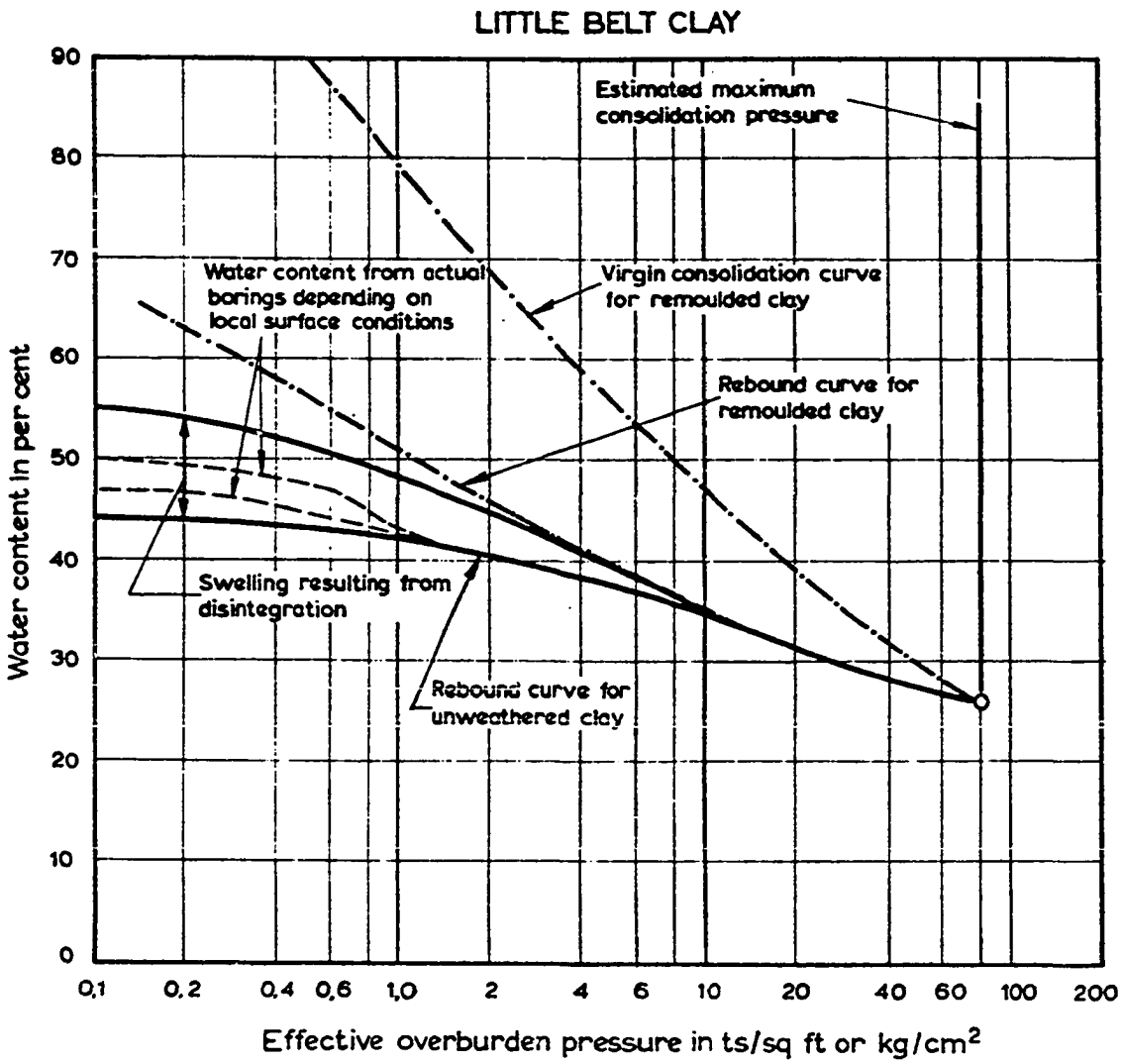


Figure5.35 Effect of weathering on consolidation characteristics of Little Belt clay, after Bjerrum (1967).

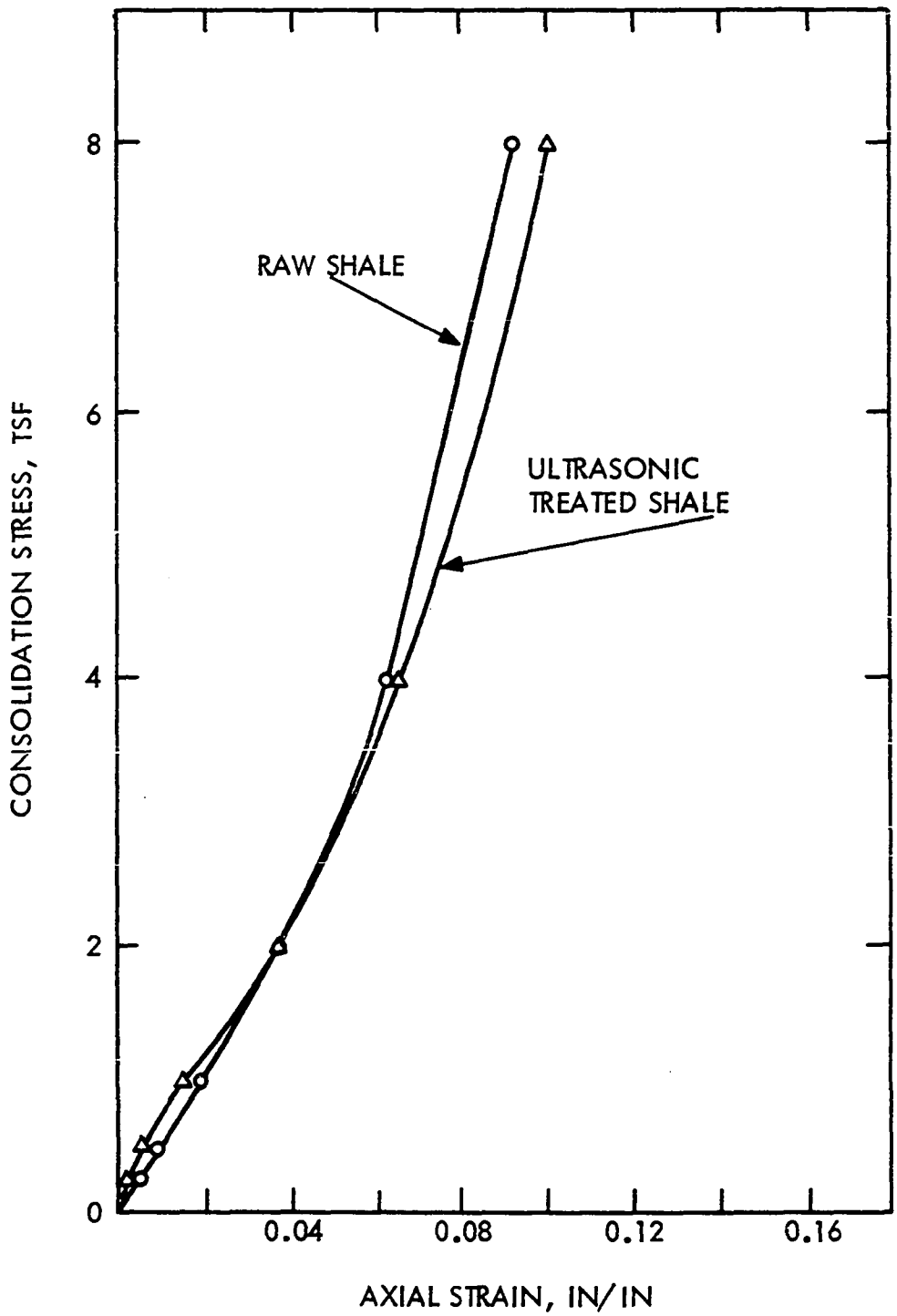


Figure 5.36 Effect of ultrasonic treatment on consolidation stress-strain relationship for shale 13.

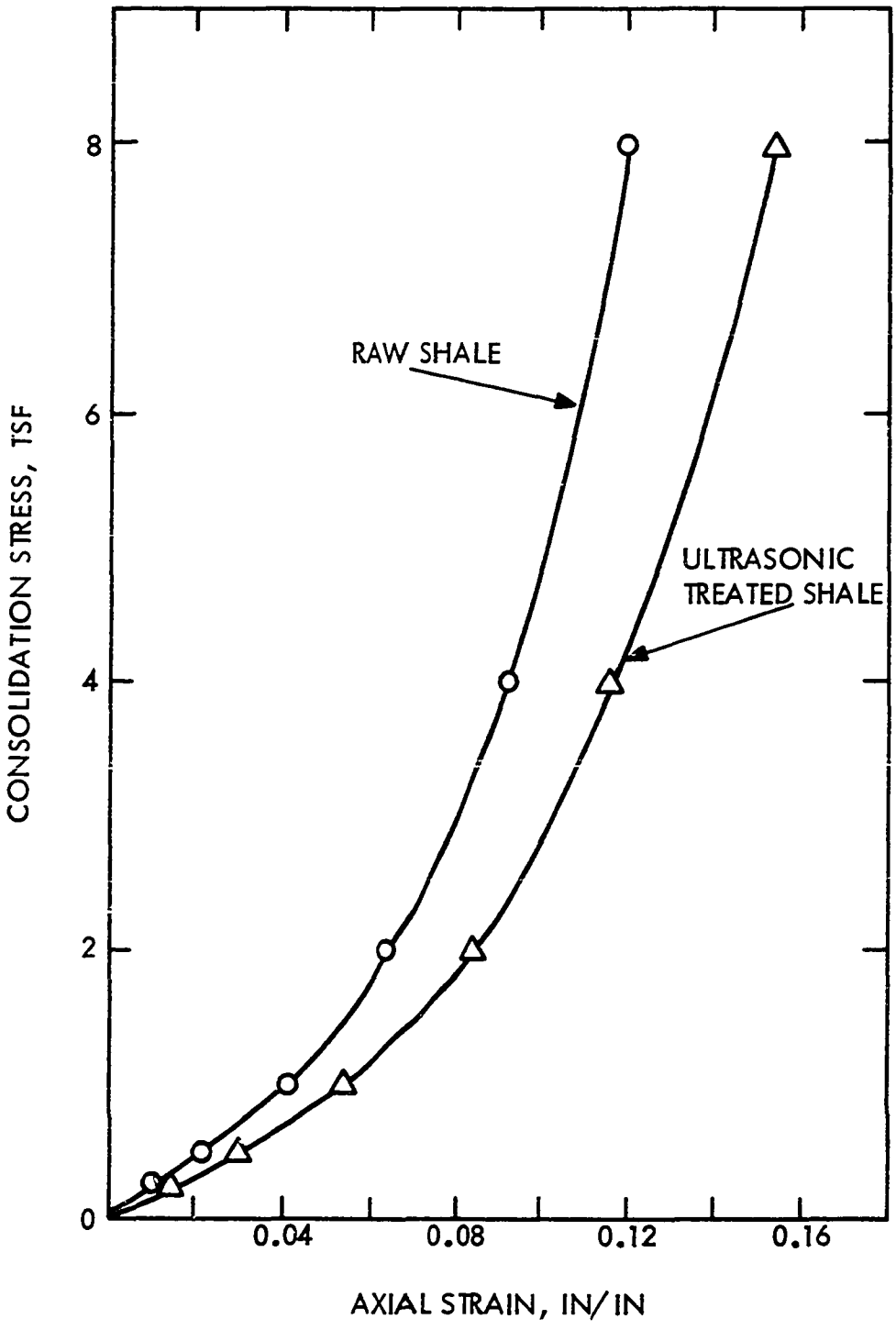


Figure 5.37 Effect of ultrasonic treatment on consolidation stress-strain relationship for shale 24.

moisture content and dry density values were almost the same after ultrasonic treatment. The insensitivity of this shale to ultrasonic treatment was also reflected in consolidation stress-strain relationships. Except for shale 13, the stress-strain curves for ultrasonic treated shales were significantly different from those for raw shales.

In the case of triaxial and unconfined compression tests, the stress-strain relationships were different with stress reaching a maximum value and showing either decrease or no increase with increase in strain and becoming approximately parallel to the axis of strain.

In consolidation stress-strain relationships, at a given strain level, the stress for ultrasonic treated shale sample was less than the stress for the raw shale samples. In triaxial tests also, the shear stress, at a particular strain level, reduced after ultrasonic treatment.

Anessi (1970) studied the consolidation stress-strain relationships of lime stabilized shales. A typical stress-strain curve is shown in Figure 5.38. At a given strain level, the stress for lime stabilized shale was higher than that of raw shale. A comparison of the pattern with the stress-strain relationship for ultrasonic treated shale indicated that the stress at the particular strain level under the same confining condition was a function of the durability of the material. For any given shale, lime stabilization and ultrasonic treatment changed its durability conditions to either extremes.

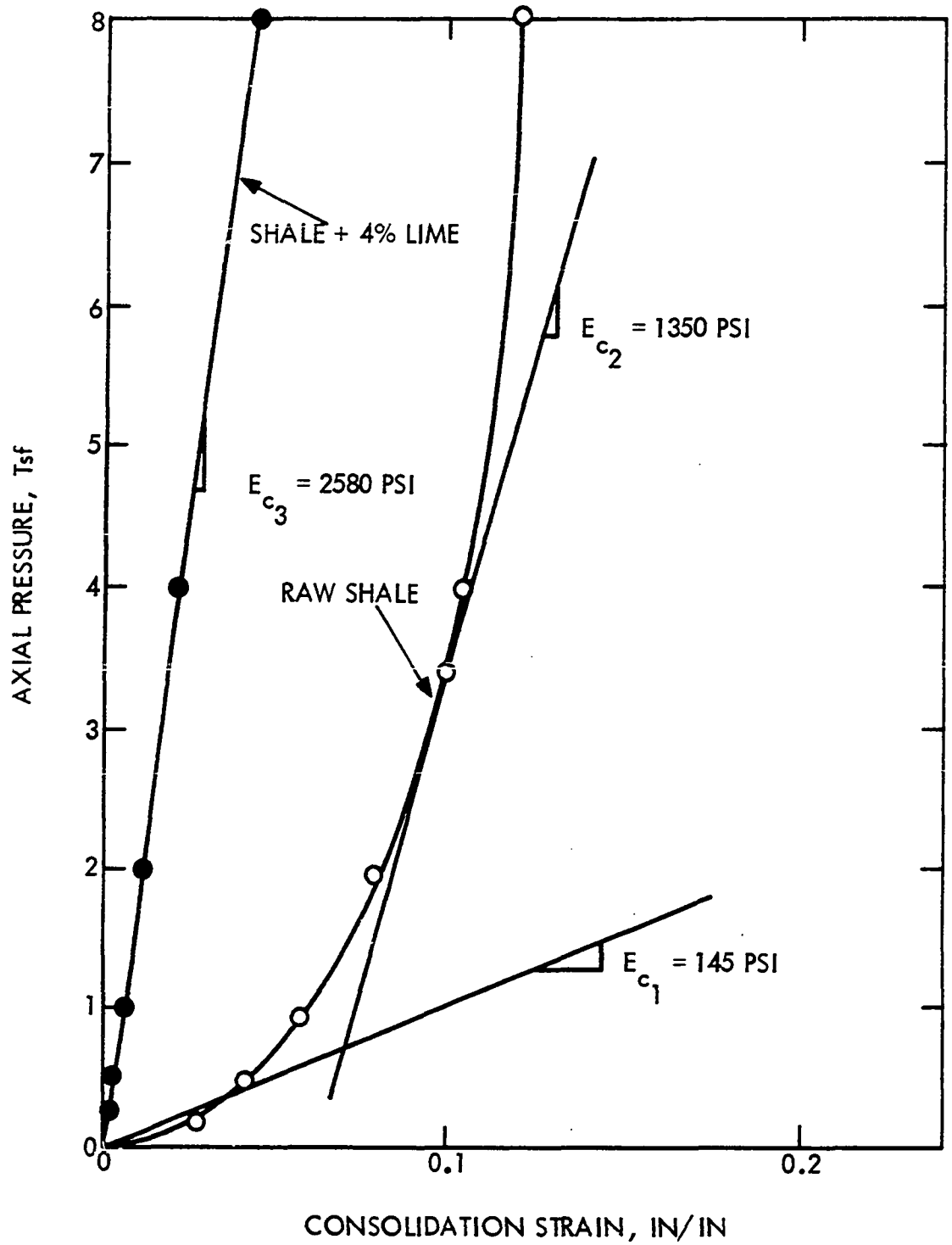


Figure 5.38 Consolidation stress-strain relationships for shale 24, after Anessi (1970).

The coefficients of consolidation and permeability were computed from the results of consolidation tests and presented in Table 5.15. The values of both parameters decreased after treatment. Both the amounts of compression during loading and decompression during unloading increased as a consequence of ultrasonic treatment. Decompression-Compression Ratio (DCR) also generally increased for all shale samples except shale 21 for which DCR decreased from 0.415 to 0.3615 after ultrasonic treatment.

There are two different mechanisms controlling the volume change behavior of saturated clays and clay shales. One mechanism is physicomechanical in nature and it governs the volume change behavior of non-expanding lattice type clays like kaolinite. The other mechanism which is physico-chemical in nature, controls that of the expanding lattice type clays like montmorillonite (Olsen and Mesri, 1970; Sridharan and Venkatappa Rao, 1973).

Each shale, in this investigation, contained more than one type of the clay minerals. Hence the volume change of these shales during consolidation was possibly controlled by both mechanisms simultaneously. However, the relative contributions of these mechanisms and any possible synergistic effects are not known.

X-Ray Diffraction Analysis

Clay mineral analysis by x-ray diffraction is generally qualitative. However, approximate proportions of clay

minerals in a sample are usually estimated from the relative intensities of the peaks on an x-ray diffraction pattern (Gillot, 1969).

X-ray diffractograms for both raw and ultrasonic treated shales are redrawn to different scale and shown in Figures 5.39 through 5.42. The locations of the peaks which are characteristics of the type of clay minerals, remained the same for ultrasonic treated shale samples. However, the peaks for ultrasonic treated shale samples appeared more defined than the patterns for raw samples. This suggests that ultrasonic treatment effectively disaggregates the particles as only aggregations cause weakened x-ray reflections.

The effect of ultrasonic treatment on the engineering properties of shale samples tested is summarized in Table 5.16. Regarding the effects of ultrasonic treatment, the modifications in consolidation and volume change characteristics might lead to the conclusion that some physicochemical changes could have taken place. But even so, these physicochemical changes could only be due to the increase in the amount less than 2 micron clay and clay size particles, as no change in chemical environment such as leaching or addition of salts occurred during treatment. The possible changes during treatment are the mechanical breakdown of aggregated clay and silt particles and abrasion of silt grains which result in increase of fines with fresh surfaces. The results of x-ray analysis and other tests for engineering properties also

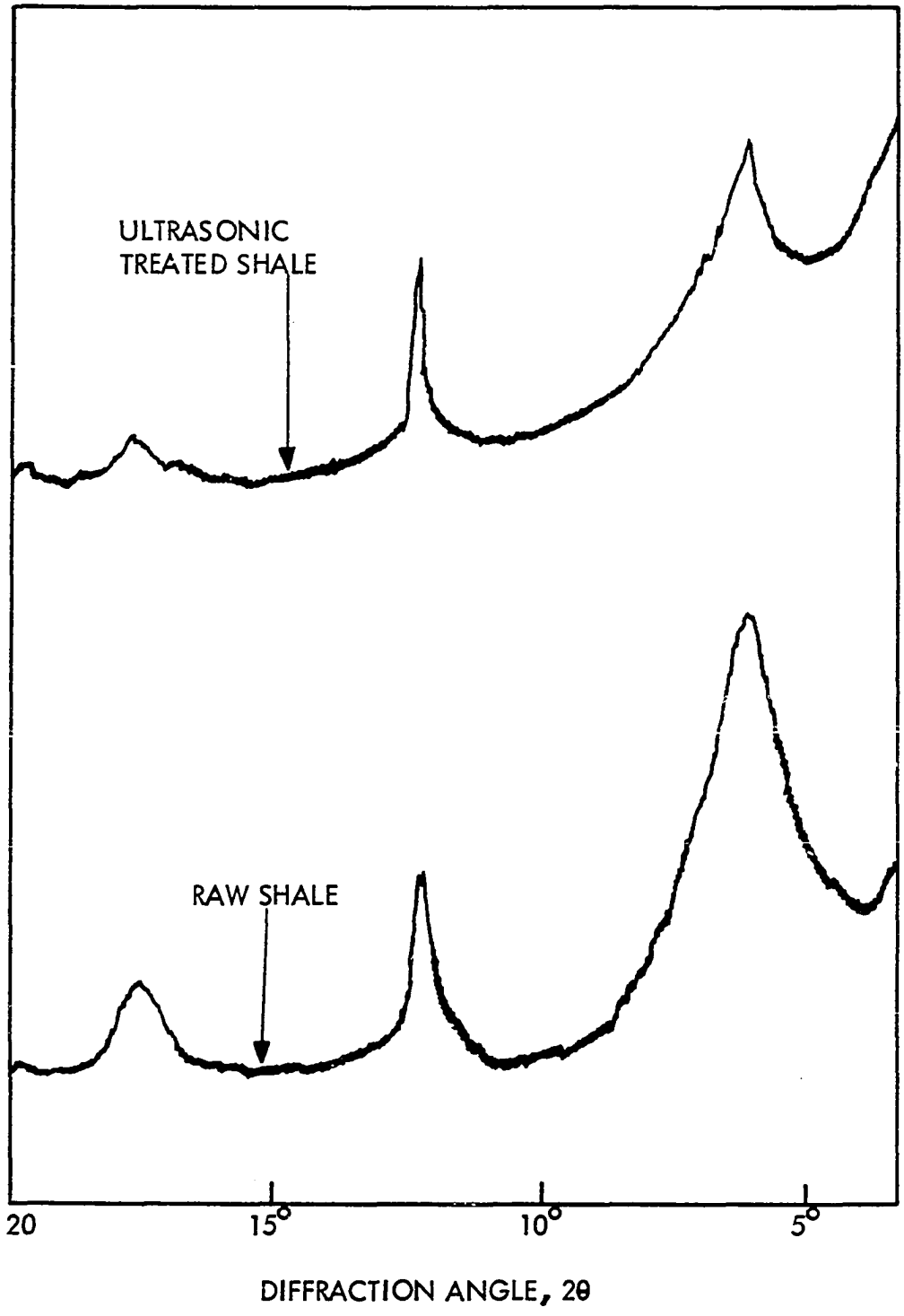


Figure 5.39 X-ray diffractograms for shale 13.

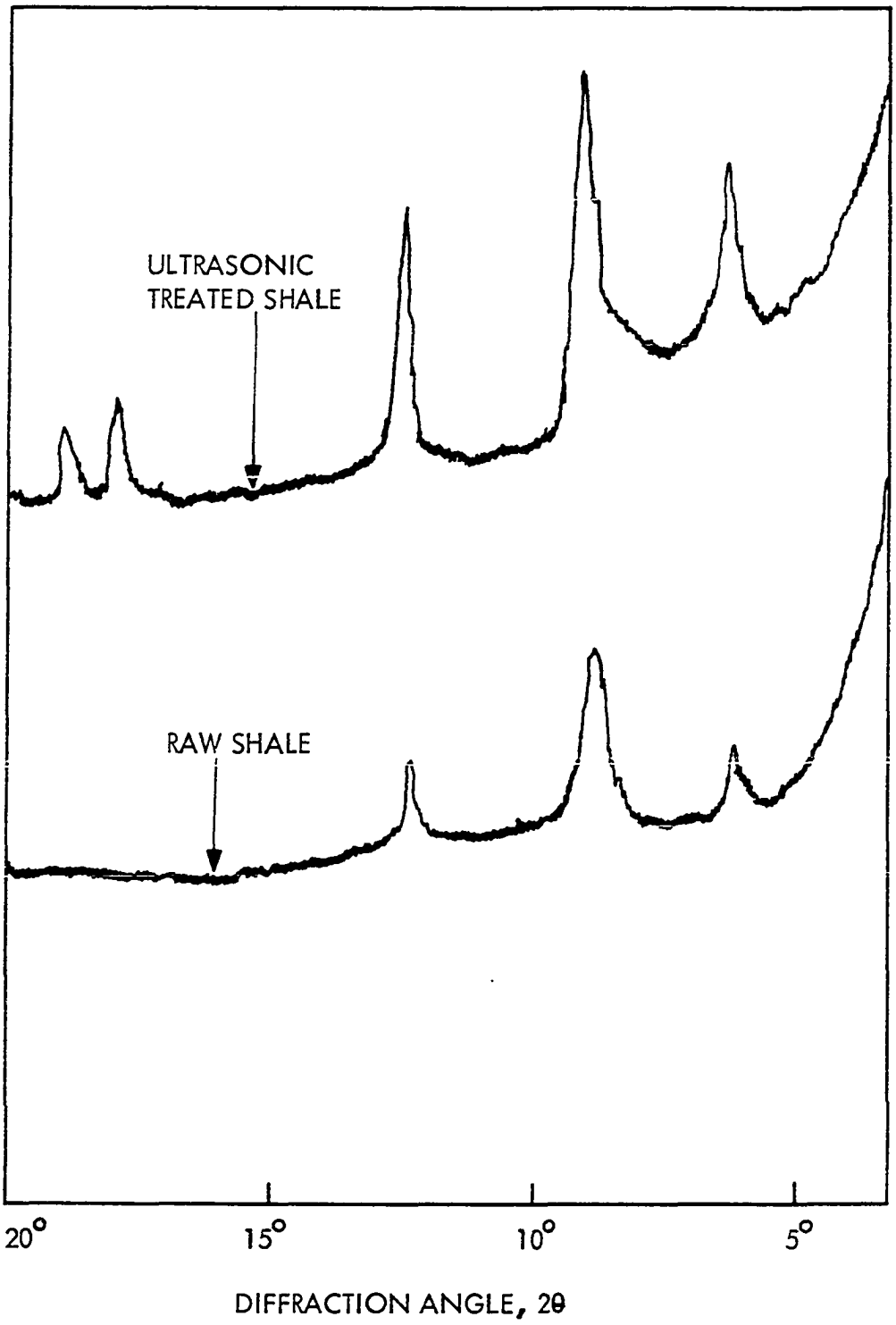


Figure 5.40 X-ray diffractograms for shale 15.

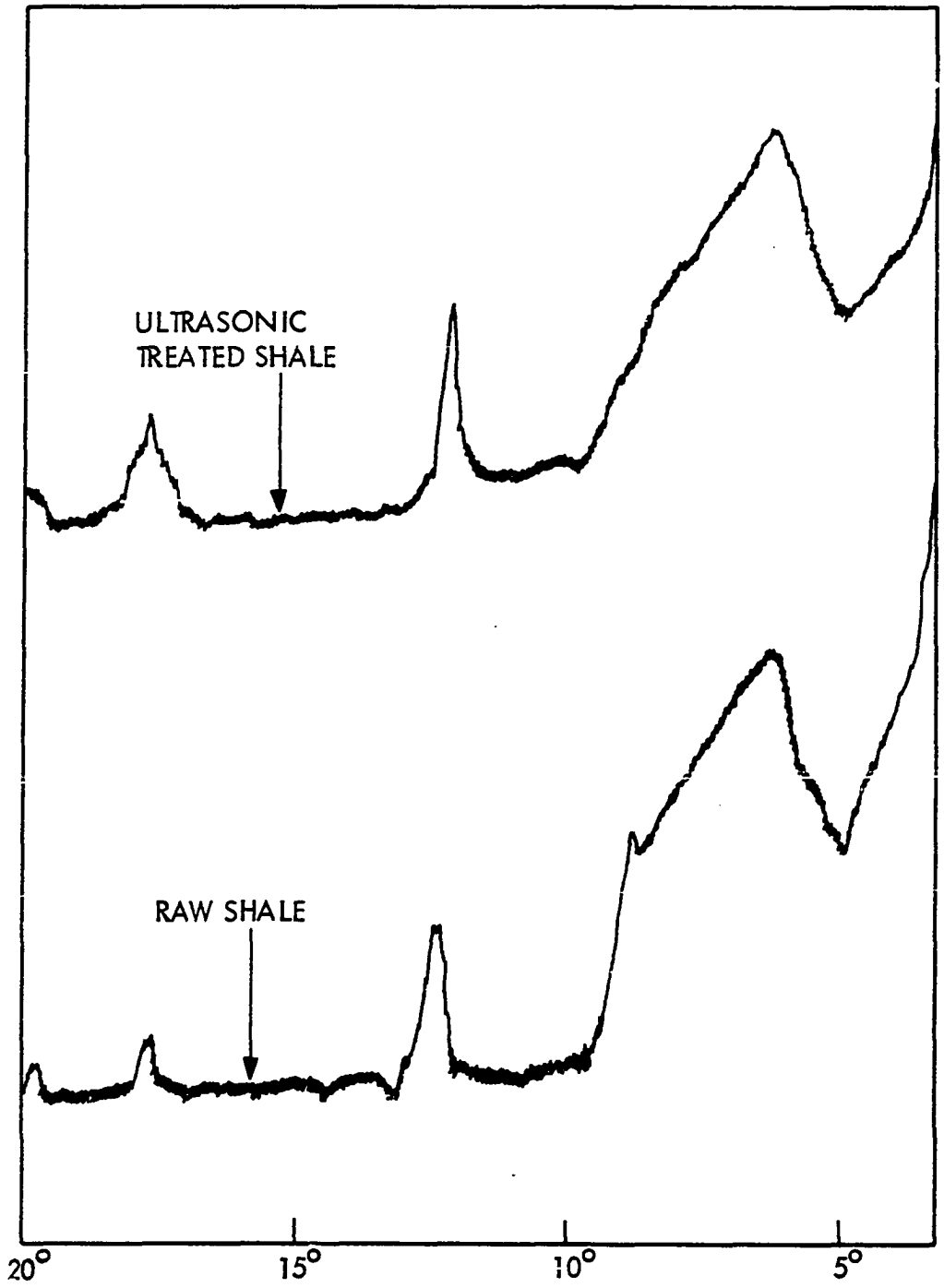


Figure 5.41 X-ray diffractograms for shale 21.

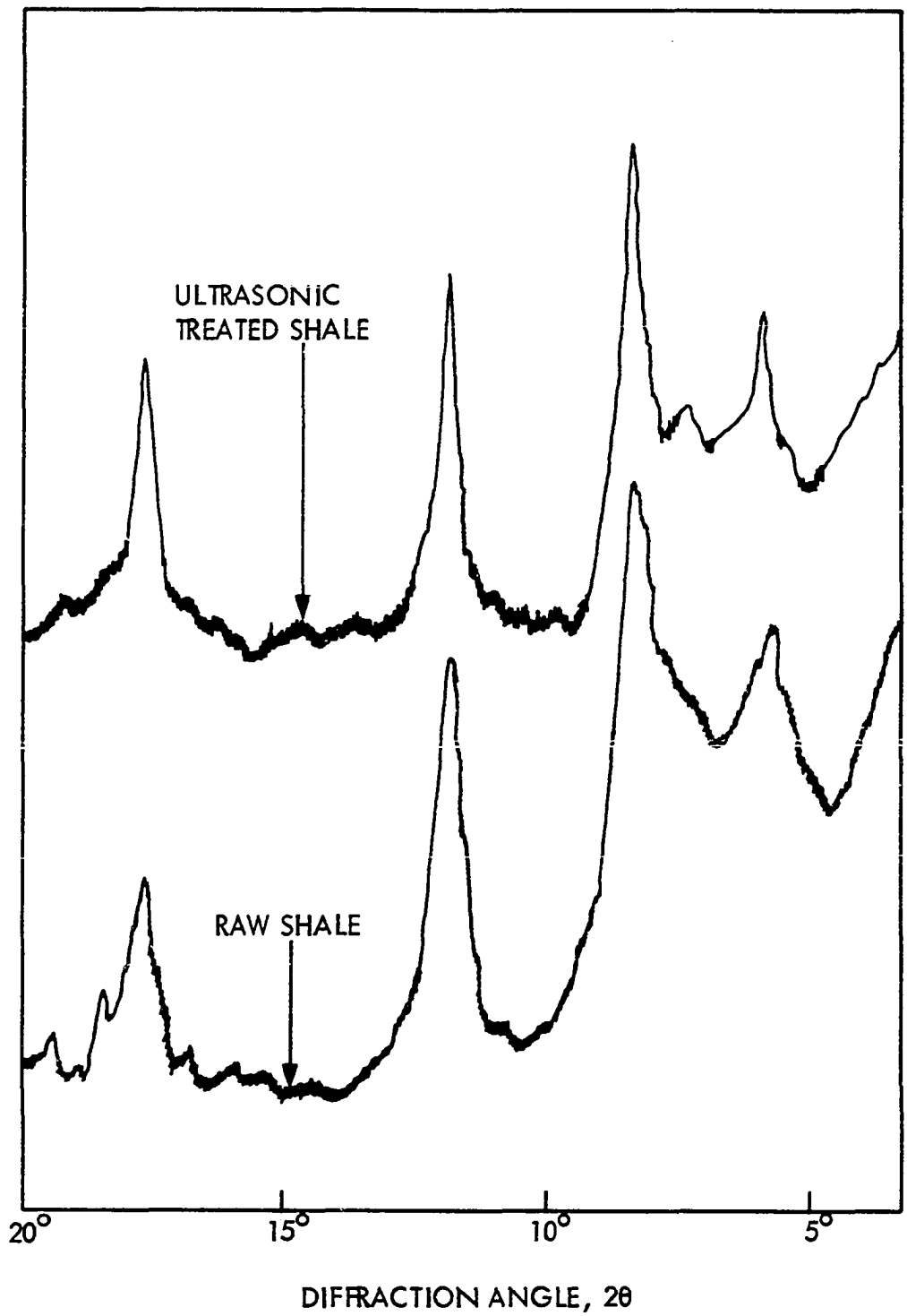


Figure 5.42 X-ray diffractograms for shale 24.

TABLE 5.16

THE EFFECT OF ULTRASONIC TREATMENT ON THE ENGINEERING PROPERTIES OF THE SHALE SAMPLES

Engineering Property	Effect of Ultrasonic Treatment
Gradation	Increase in the amount of fines.
Atterberg Limits	Increase in the values of liquid limit and plasticity index.
Specific Gravity	No change.
Moisture-Density Relationships	Decrease in density and increase in the amount of optimum moisture content.
Shear Strength	Decrease in strength. At the same water content, strength was higher.
Relaxation	No change except that the deviatoric stress was affected.
Volume Expansion	Significant increase in illitic shales.
Consolidation	Compressibility increased. Permeability decreased.
Mineralogy	No change.

indicate that the effect of ultrasonic treatment is primarily reflected in the increase of fines and hence concluded to be physicomechanical.

Disaggregation Mechanism of Ultrasonic Treatment

It was found that, for a given amount of treatment, the shales disaggregated in varying amounts. In this section, an attempt is made to explain the response differences of the shales to ultrasonic treatment.

Method of Analysis

The composition of shales consists usually of clay minerals, quartz and feldspar; and sometimes free salts and organic matter. All these minerals are either pure silicates or silicates of Al, Fe, Ca, Mg, K or Na. Although the mineral composition of all shales is basically the same within a definite range, durability characteristics may be different depending on the geologic past and degree of weathering the shales have been subjected to. During the process of weathering, the cementation of minerals is broken and some of the silicates react with carbonic acid to form water soluble salts, resulting in disintegration of shales.

Chandler (1972) reported that with progress of weathering, the amount of calcium carbonate decreased and the amount of ferric oxide increased. It is also known that ion exchange takes place with substitution of one metallic ion for the other. Hence the mineralogical composition seems to undergo

perpetual changes with weathering processes. Although it is known that the chemical composition varies for a given mineral and the same chemical composition may be obtained for different minerals, it is still possible that the changes in the mineralogy during weathering may be reflected, at least to a certain degree, in the chemical composition. Hence chemical composition is employed to explain the response differences of shales to ultrasonic treatment.

Data on chemical analysis of Oklahoma shales obtained by x-ray fluorescence technique are presented in Table 5.17. The values indicated for each compound include the chemical composition of clay minerals, non-clay minerals and free salts. Based on prior experience, some of these data appear to assume values higher than expected. The chemical analyses were not, however, rerun. Consequently, the discussion which follows should be viewed with this limitation in mind.

For sedimentary rocks including shales, typical values for oxides of Fe, Mg, K and Na are about 2 to 3 percent and for Ca about 5 percent. Any values higher or lower than these values would indicate either the scatter or the extent of ion exchange activity and presence of excess salts which are the results of weathering. For Oklahoma shales the amount of Fe_2O_3 varied from 5.19 to 14.86 percent and for CaO from 0.43 to 14.83 percent. These variations may indicate the degree of weathering and hence the resistance of shales to ultrasonic treatment. Also indicated in Table 5.17 are the

TABLE 5.17

CHEMICAL COMPOSITION AND PARAMETERS QUANTIFYING THE EFFECT OF ULTRASONIC TREATMENT FOR SHALE SAMPLES

Shale No.	DI*	2μ **	PI ***	pH	SiO_2	Al_2O_3	Fe_2O_3	MgO	CaO	Na_2O	K_2O
6	0	0	0	7.7	55.86	18.50	11.76	1.65	1.18	0.04	1.49
7	13	12	5	7.8	59.70	16.28	7.41	2.59	3.50	0.75	3.82
8	21	21	-	6.0	66.95	14.30	5.99	2.26	0.63	0.05	4.77
9	13	12	3	8.4	59.70	16.71	9.18	2.50	2.68	1.05	3.18
10	13	12	0	7.9	60.55	18.77	8.03	1.41	1.73	0.98	2.38
11	4	3	8	7.7	53.09	13.65	5.19	2.36	14.83	0.29	2.19
11A	10	7	18	7.5	56.28	15.21	5.63	2.52	9.98	0.56	2.36
12	47	14	2	7.8	57.14	17.85	9.72	2.04	3.30	0.18	3.21
13	19	10	9	5.1	65.88	16.19	11.40	0.84	0.91	0.08	0.76
14	29	27	1	6.8	57.35	19.75	9.63	2.08	0.56	0.22	3.76
15	55	47	18	7.9	57.78	17.05	10.60	3.03	0.77	1.75	3.48
16	43	37	4	8.6	58.20	16.79	10.43	3.42	0.97	1.53	4.15
17	31	25	4	8.5	55.65	15.70	9.72	5.00	2.00	0.45	3.78
18	62	26	10	8.1	56.71	16.33	9.36	5.73	0.50	0.68	4.88
19	65	46	3	8.2	55.01	16.29	10.87	4.38	1.25	0.49	4.17
20	21	13	9	9.4	59.06	16.86	13.00	2.58	0.43	2.58	0.81
21	49	37	9	8.5	58.63	16.41	11.76	3.09	1.11	3.09	0.51
22	38	14	10	8.4	53.94	19.49	13.62	1.93	1.11	0.18	1.42
23	33	22	6	7.5	60.76	17.17	9.10	2.07	0.56	0.64	3.27
24	59	51	16	7.6	50.31	17.75	14.86	1.43	2.20	0.27	3.50

*Column 2: Disaggregation Index.

**Column 3: Increase in Amount Finer then 2 micron.

***COlumn 4: Increase in Plasticity Index.

values of increases in the amount of 2 micron size particles and plasticity index. Using these data, a statistical analysis was performed.

Choice of Parameters

For quantitative assessment of the effect of ultrasonic treatment, the changes in gradation and plasticity index seemed to be suitable parameters. However, these parameters do not indicate the amount of fines or clay size particles. This is very important because the amount less than 2 micron size particles would relate to the degree of weathering of raw shales. Therefore another parameter termed "Disaggregation Index" was developed and introduced in the analysis. It was calculated as follows:

$$\text{Disaggregation Index (DI)} = 100\ (b-a)/(100-a)$$

where a = amount less than 2 micron size in raw shale sample

b = amount less than 2 micron size in ultrasonic treated shale sample

Using these values, a disaggregation index chart is also devised and shown in Figure 5.43 with the values of disaggregation index for all shales.

Regression analyses were run using one of the above three parameters as dependent variable and the values of soil pH and chemical compositions as independent variables. Semi-logarithmic relationships were also attempted. The values of

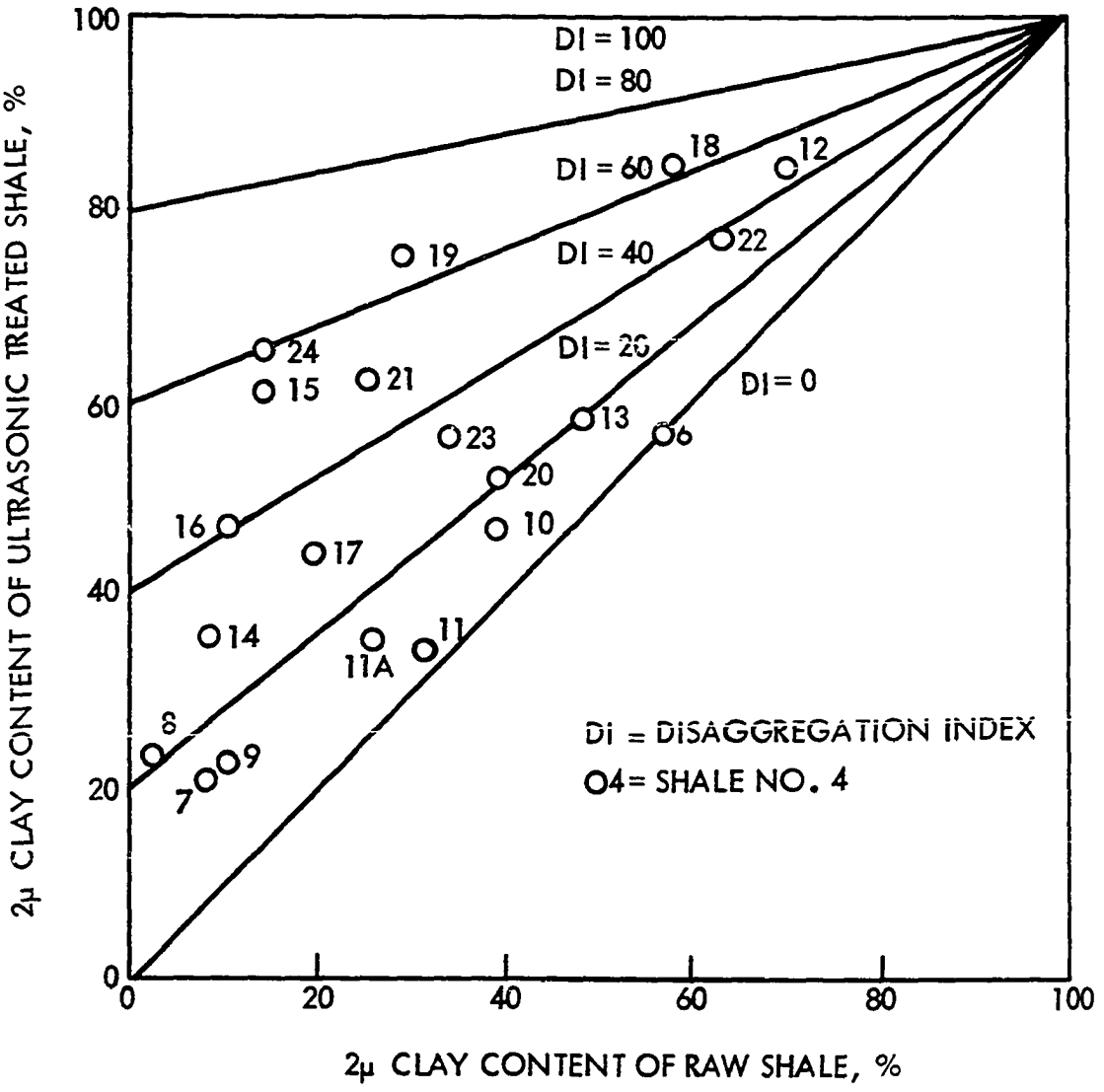


Figure 5.43 Disaggregation index chart showing the disaggregation indices for Oklahoma shales.

correlation coefficients, regression coefficients, multiple correlation coefficients and standard errors of estimate are summarized in Table 5.18. The parameter, amount less than 2 micron clay and clay size particles, had the best multiple correlation coefficient. Both normal and semi-logarithmic regression equations gave more or less the same value with a standard error of estimate of 8.

Results

As shown in Table 5.18, the correlation coefficients obtained among various variables generally ranged from 0.027 to 0.728. For each indicative parameter, the correlation coefficients between the parameter and other variables were compared.

For the parameters PI(+) and log PI(+), the correlation coefficients between one of these parameters and other compounds ranged from 0.027 to -0.346. The poor correlation indicated a wide scatter. This was probably due to the operator dependency of the limit tests. These parameters will not be considered for further discussion.

For the parameters 2μ(+), log 2μ(+), DI and log DI, the correlation coefficients ranged from 0.108 to -0.728. Admittedly, the correlation coefficients were low but on a relative scale it is possible to arrange them in an order of magnitude which also displays the order of significance of each compound as indicated in Table 5.19.

TABLE 5.18

PROPERTY INTERRELATIONSHIPS BETWEEN THE CHEMICAL COMPOSITION AND CHANGES IN INDEX PROPERTIES AFTER ULTRASONIC TREATMENT FOR ALL THE SHALES

Dependent	A_o	pH	SiO_2	Al_2O_3	Fe_2O_3	MgO	CaO	Na_2O	K_2O	r[b]	SSE[c]
2 (+)[a]	--	0.162	-0.313	0.164	0.524	0.340	-0.463	0.273	0.489	--	--
2 (+)	504.69	-7.204	-4.888	-7.409	-0.600	-3.924	-5.467	2.690	1.538	0.918	7.790
Log(2 (+))	--	0.153	-0.127	0.288	0.567	0.378	-0.705	0.239	0.554	--	--
Log(2 (+))	8.632	-0.106	-0.076	-0.112	-0.009	-0.047	-0.116	0.160	0.053	0.940	0.149
DI	--	-0.141	-0.377	0.281	0.595	0.495	-0.500	0.117	0.417	--	--
DI	216.83	-7.835	-2.637	-1.345	3.153	4.678	-2.101	5.239	3.618	0.877	12.390
Log(DI)	--	0.188	-0.180	0.425	0.674	0.370	-0.728	0.108	0.394	--	--
Log(DI)	4.02	-0.094	-0.037	-0.015	0.036	0.057	-0.061	0.034	0.041	0.914	0.177
PI(+)[a]	--	-0.070	-0.233	-0.171	0.122	-0.143	0.203	0.182	-0.271	--	--
PI(+)	222.63	-3.143	-2.132	-2.365	-1.095	-0.624	-1.707	8.392	-5.029	0.761	4.890
Log(PI(+))	--	0.027	-0.138	-0.336	0.141	-0.069	0.196	0.173	-0.345	--	--
Log(PI(+))	15.90	-0.119	-0.136	-0.246	-0.080	-0.066	-0.142	0.428	-0.360	0.789	0.301

[a]Increases in either the amount less than 2 micron or plasticity index.

[b]Multiple correlation coefficient.

[c]Standard error of estimate.

TABLE 5.19

RELATIVE IMPORTANCE OF CHEMICAL COMPOUNDS GOVERNING THE DISAGGREGATION MECHANISM

Indicative Parameter	Chemical Compounds[a]	Ranking of Chemical Compounds[b]					
		Fe_2O_3	CaO	K_2O	MgO	Al_2O_3	SiO_2
$2\mu(+)$[c]	Fe_2O_3, K_2O, CaO, MgO, SiO_2	5	3	4	2	0	1
Log $2\mu(+)$	CaO, Fe_2O_3, K_2O, MgO, Al_2O_3	4	5	3	2	1	0
DI	Fe_2O_3, CaO, MgO, K_2O, SiO_2	5	4	2	3	0	1
Log DI	CaO, Fe_2O_3, Al_2O_3, K_2O, MgO	4	5	2	1	3	0
Total		18	17	11	8	4	2

[a]Arranged in the order of magnitude of correlation coefficients.

[b]Values assigned in the order of occurrence.

[c]Increase in amount less than 2 micron size particles.

To quantify the relative importance of the five components as indicated by these four parameters, a ranking scheme was followed. Each compound was assigned a numerical value depending on the order of its occurrence. For example, considering the variable $2\mu(+)$, Fe_2O_3 would be assigned a value of 5; K_2O, 4; CaO, 3; and so on. This procedure was followed for each variable and the total value for each compound was computed. The calculations are shown in Table 5.19. The components, influencing the disaggregation mechanism, in the order of importance are Fe_2O_3, CaO, K_2O, MgO, Al_2O_3 and SiO_2.

Further, the results of the statistical analysis lead to a number of observations which can be utilized to explain the disaggregation mechanism of ultrasonic treatment. As explained earlier, the chemical data lack the depth required in statistically designed laboratory experimentation. Thus, the relationships presented below should be viewed not as having a quantified exactitude, but rather displaying trends and tendencies.

1. Low values of correlation coefficients (0.106 to 0.188) obtained for soil pH with other variables indicated that soil pH had very little or no influence of the disaggregation of shales during ultrasonic treatment as no leaching or addition of salts was possible in the tank type equipment.

2. The compound Fe_2O_3 was of major significance. Generally, the amount of Fe_2O_3 in a soil could be either free Fe_2O_3 or oxidized FeO or both. The degree of oxidation of FeO is often related to the degree of weathering. In the present analysis, the amount of FeO was not determined separately but it was included in the Fe_2O_3 observed by x-ray fluorescence. This accounts for the high values of Fe_2O_3 for certain samples. However, the statistical analysis indicated fairly high values of positive correlation between the amount of Fe_2O_3 and the parameters quantifying disaggregation suggesting that the disaggregation increased proportionally with the amount of Fe_2O_3. If high values of Fe_2O_3 were entirely due to the presence of Fe_2O_3, then it would be possible to conclude that the shale sample was already weathered. On the other hand, when high values of Fe_2O_3 reflect the presence of "unoxidized" FeO, the shale is said to have somewhat weathered and also the potential of being weathered. No distinction can be made, however, between the degree of weathering reached and that which ultimately can be attained. In either case, for shale samples with high values of Fe_2O_3 the resistance to ultrasonic treatment was low.
3. The correlation coefficients for CaO varied from -0.463 to -0.728. CaO seemed to resist the breakdown in ultrasonic treatment as the amount of CaO correlated negatively with the parameters chosen to quantify the effect of

ultrasonic treatment. However, the amount of CaO found in the chemical analysis might include calcium ions substituted as cations in the clay minerals and other free calcium salts which provide a good cementing agent when present in the soil. A typical example is calcium carbonate which also accounts for the strength improvement in the soils after the addition of lime.

4. Among other important compounds, the compounds K_2O, MgO, and Al_2O_3 indicated positive correlation and SiO_2 negative correlation. It may be speculated that the positive correlation of K_2O and MgO would probably suggest the lack of cementing properties of salts of these metals and that of Al_2O_3 would suggest weak cation linkage. Negative correlation of SiO_2 indicated that the disaggregation tendency would decrease with increase in the amount of silica.
5. Each of the chemical compounds correlated poorly with the parameters quantifying disaggregation of shales. But, when all compounds were considered collectively along with soil pH high values of multiple correlation (0.877 to 0.940) for the regression line were obtained. This would imply that the mechanism of ultrasonic breakdown was actually controlled by the mineralogical composition of shales and the mineralogical composition was indirectly but completely described by the overall chemical composition. However, the better correlation with the chemical

composition indicated that the mechanism of ultrasonic disaggregation was closely related to the overall chemical composition of raw shales with Fe_2O_3 and CaO being the important compounds.

Usefulness of Ultrasonic Treatment

With the knowledge of the chemical composition, the parameters quantifying the disaggregation tendency and in turn the weatherability of shales could be predicted. However, obtaining the chemical data would be an expensive method. On the other hand, ultrasonic treatment is simple and economical to perform and indicates the weatherability of shales.

Also, the shale materials, used in pavements are not only exposed to weathering agents but are continuously subjected to traffic induced stresses. Chemical changes would be far less for the materials under pavement than for the materials in natural state since the life of pavement is negligibly short on a geological time scale. Moreover, the shale materials used in pavements usually have protective coverings. Hence it seems that the material breakdown of the shales is similar to the breakdown during ultrasonic treatment and ultrasonic treatment could be used to simulate field weathering.

CHAPTER VI

CONCLUSIONS

This investigation encompasses the study of the effect of ultrasonic treatment on Oklahoma shales and it is divided into two parts. In the first part, four Oklahoma shales varying in clay mineral type and content were subjected to ultrasonic treatment and their engineering properties were determined before and after treatment. In the second part, using the data on 24 Oklahoma shales which exhibit different degrees of weathering, useful relationships among some of the engineering properties of shales were obtained, to provide a predictive tool in the behavior of shale after ultrasonic treatment.

On the basis of the data obtained in the first part, the following conclusions are drawn:

1. Ultrasonic treatment disaggregated the clay shales and compact shales very effectively and increased the amount of clay and clay size particles in direct proportion to their weatherability.
2. The change in textural properties of the shales after ultrasonic treatment was reflected in the plasticity

characteristics increasing both the values of liquid limit and plasticity index.

3. As a result of ultrasonic treatment, the maximum dry density decreased and the amount of optimum moisture content increased. The weathering processes would also reduce the stability of the materials resulting in low density and high natural moisture content. Hence ultrasonic treatment altered the moisture-density relationships in a way similar to the natural weathering processes.
4. The molding moisture content influenced the strength properties of the compacted shales specimens during triaxial testing. The higher the molding water contents, the lower were the strength parameters irrespective of the rates of strain applications.
5. Ultrasonic treatment reduced the shear strength in a way similar to the natural weathering. However, at the same water content, the ultrasonic treated shales showed higher strength than that of raw shale.
6. The relaxation characteristics did not seem to be affected by ultrasonic treatment as the curves for both raw and ultrasonic treated shales were almost the same.
7. The rate of strain application had considerable effect on the relaxation characteristics, reducing the percent relaxation with lower rates of strain.
8. Volume change characteristics were modified after ultrasonic treatment. Of the shales tested, increase in volume

change as a result of ultrasonic treatment was more for silty shales than for clay shales. This is primarily due to the large increases in the amount of clay and clay size particles and consequent surface hydration of these particles.

9. The values of coefficient of consolidation and coefficient of permeability generally reduced after ultrasonic treatment. The e-log p relationships became steeper, both during loading and unloading cycles, in a manner similar to that of naturally weathered clays and clay shales.
10. The results of x-ray diffraction analyses indicated that there was no change in the types of the clay minerals after ultrasonic treatment. However, due to the disaggregation processes, the peaks of the identified clay minerals became well defined.
11. A comparison of the engineering properties and x-ray analysis before and after treatment indicated that the effects of ultrasonic treatment are primarily physico-mechanical.
12. Ultrasonic treatment is a simple and fast method of simulating weathering, and it is very useful for studying the effects of weathering on the pavement materials. It can serve as a predictive test for testing the suitability of shales for use in highway construction.

On the basis of the results of the statistical analysis obtained in the second part, the following conclusions are presented.

13. Statistical analyses of the test data for Oklahoma shales showed that the reaction potential or plasticity index correlated well with volume change as these parameters characterized both the type and amount of clay minerals.
14. The reaction potential or plasticity index could be used either as single parameters or along with dry density and moisture content to predict the volume change potential of the compacted shales within an accuracy of 3 percent.
15. Statistical analyses of the data on chemical composition and index properties of raw and ultrasonic treated shale samples showed that each of the compounds correlated poorly with the parameters quantifying disaggregation. However, good correlation was obtained when all chemical compounds were considered. This observation implied that the disaggregation mechanism was controlled by the mineralogical composition which was completely described by the total chemical composition.
16. The analyses further indicated that Fe_2O_3 and CaO correlated better than other compounds. The amount of Fe_2O_3 increased with increase in weatherability of the shale. The increases in Fe_2O_3 could be correlated to the degree

of weathering and used for determining the durability of the shales. Disaggregation of the shales was found to vary inversely with the amount of CaO which contributes to the durability of the shales.

CHAPTER VII

RECOMMENDATIONS FOR FURTHER STUDY

In this study, the mechanism of ultrasonic treatment and its effect on the engineering properties of compacted shales were studied. Also, method of ultrasonic testing in simulating natural weathering conditions and its usefulness as an identification test in determining the suitability of shales have been outlined. However, for establishing more definite test criteria, the following are recommended for further work.

1. For a given shale since weathering decreases with depth, samples obtained from different depths should be tested. The effect of ultrasonic treatment then for a given weathering process can be correlated to the progress of weathering with depth. Data on chemical composition of shales will help explain the role of one or more mineralogical constituents in resisting or accelerating a particular weathering process. Later on this may be extended to samples from zones of different weathering processes and known goelogical history.
2. Shale samples from the highway construction projects should be obtained at periodic intervals, starting from

the time of opening the highway to traffic, to study the effects of weathering and traffic stresses on the durability of the subbase and subgrade materials. The data obtained from the ultrasonic testing for different durations of treatment then can be correlated to the field data. This would help decide the test criteria for material suitable for different types of highway pavements.

3. Shales with kaolinite and chlorite as predominant clay minerals should be included in the study to understand the effect of different clay minerals on the ultrasonic disaggregation.

4. In the present investigation, the data on 24 shale samples were considered for statistical analyses. Additional test data on ultrasonic treatment, index properties, strength and volume change characteristics and chemical composition would be valuable in obtaining more reliable relationships for predicting weatherability, volume change and other index properties.

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