

Removal of Water and Rearrangement of Particles During the Compaction of Clayey Sediments—Review

GEOLOGICAL SURVEY PROFESSIONAL PAPER 497-B



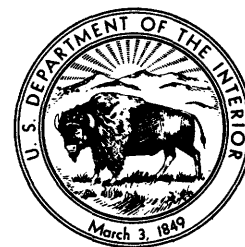
Removal of Water and Rearrangement of Particles During the Compaction of Clayey Sediments—Review

By ROBERT H. MEADE

MECHANICS OF AQUIFER SYSTEMS

GEOLOGICAL SURVEY PROFESSIONAL PAPER 497-B

Review of the pertinent literature on the factors influencing the water content and clay-particle fabric of clayey sediments under increasing overburden pressures



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GLOSSARY

Compaction. “* * * decrease in volume of sediments, as a result of compressive stress, usually resulting from continued deposition above them” (American Geological Institute, 1957, p. 58). This process is called consolidation in the soil-mechanics literature.

Consolidation. “* * * any or all processes whereby loose, soft, or liquid earth materials become firm and coherent” (American Geological Institute, 1957, p. 62). This includes cementation as well as compaction.

Fabric. “* * * the physical constitution of a * * * [sediment] as expressed by the spatial arrangement of the solid particles and associated voids. Since * * * fabric is concerned with spatial arrangement it is described in terms of the orientation and distribution patterns of the primary particles, compound particles, and voids” (Brewer and Sleeman, 1960, p. 173).

Turbostratic. A type of fabric in which clay-mineral particles are in aggregates within which the orientation is preferred and between which the orientation is random (Aylmore and Quirk, 1960, p. 1046; adapted from Biscoe and Warren, 1942, p. 370).

Void ratio. Ratio (e) of the volume of pore space to the volume of solid particles in a sediment (American Society of Civil Engineers, 1958, p. 40). Its relation to percent porosity (n) is expressed in the following formula:

$$e = \frac{n}{1-n}$$

In all the studies discussed in this review, the void ratio is a volumetric index of the water content.

MECHANICS OF AQUIFER SYSTEMS

REMOVAL OF WATER AND REARRANGEMENT OF PARTICLES DURING THE COMPACTION OF CLAYEY SEDIMENTS—REVIEW

By ROBERT H. MEADE

ABSTRACT

Among the factors that influence the amount of water held in clayey sediments under overburden pressures between 0 and about 50 kg per cm² are particle size, clay minerals, exchangeable cations, and interstitial-electrolyte concentration. The amount of water held in clayey sediments varies inversely with particle size through a wide range of pressures and chemical conditions. The influence of different clay minerals on water content is related mainly to their particle size. The influences of exchangeable cations and electrolyte concentration also vary with particle size: in fine-grained clays (montmorillonite, for example), water content varies inversely with the valence of exchangeable cations and the concentration of monovalent electrolyte; in coarse-grained clays (kaolinite, for example), water content varies directly with the cation valence and the electrolyte concentration.

Under overburden pressures greater than about 50 kg per cm², the major influences on the water content of clayey sediments seem to be particle size, clay minerals, and temperature. As temperature increases, less pressure is needed to compact the clay. Variations in exchangeable cations and in electrolyte concentration do not seem to influence the water content under higher pressures, but they do affect the clay-particle fabric.

Although preferred and turbostratic orientations of clay-mineral particles can be produced by compressing clays in the laboratory, there is little evidence to show that they are formed readily during natural compaction. Experimental evidence suggests that preferred orientation of particles, normal to overburden pressures, would be most likely to form in sediments that have large clay-mineral particles, low concentration and low acidity of interstitial electrolyte, and low valence of exchangeable cations. The formation of a preferred orientation also seems to be favored by the presence of organic material and the existence of a partly oriented fabric at the onset of compaction. The formation of a turbostratic fabric (particles oriented in domains which are oriented at random with respect to other domains) instead of generally preferred orientation might be favored by high electrolyte concentration and high cation valence.

Among the factors whose influence on water removal and particle rearrangement might be significant, but about which little is known, are the anions and organic matter in the interstitial solutions. Also, very little is known of the relative effect of each of the several factors when their influences are combined under complex natural conditions.

INTRODUCTION

The advances in clay technology and soil mechanics within recent years—especially in the decade 1953–62—make possible a new look at the compaction of fine-grained sediments. Instead of considering compaction as a simple relation between pore volume and overburden pressure, one can now take into account the influence of other physical and chemical factors. This review outlines a current set of questions that geologists and hydrologists can ask while investigating the compaction of clayey sediments.

This is a summary of the pertinent knowledge as of 1962 of the water in clays, the removal of water under pressure, and the spatial arrangement of clay-mineral particles in sediments. Most of the information is taken from publications in fields other than geology—soil science, soil mechanics, ceramics, and physical and colloidal chemistry. Except for the theoretical discussion of forces in the first section, this review is concerned mainly with reported evidence and is only secondarily concerned with speculations and conclusions. Papers that contain little or no evidence to support their conclusions are not reviewed. Furthermore, the experimental evidence on clay behavior that has little direct counterpart in nature—the work on the influence of lithium and of cesium electrolytes, for example—is not treated fully. Finally, I have tried to point out the conspicuous gaps in the present body of knowledge.

In this review I assume that the reader is already generally familiar with the structure and composition of the common clay minerals and with their more distinctive properties, such as minute size, large surface area, platy habit, and ion-adsorption and ion-exchange capacity. For a review of these subjects, see the recent and concise summary by Grim (1962, p. 7–51) or the earlier standard works by Grim (1953) and by Jasmund (1955). I also assume that the reader is already familiar with the general pattern of the reduction of pore volume of clays under increasing overburden loads,

which was treated in the classic paper by Hedberg (1936) and which was reviewed more recently by Engelhardt (1960, p. 35-50), by Lomtadze (1955, 1956) and by Weller (1959, p. 274-294).

PURPOSE OF STUDY

The U.S. Geological Survey is making an investigation directed toward determining the principles controlling the compaction of aquifer systems resulting from change in grain-to-grain load (effective stress) induced by major decrease of internal fluid pressure. This project is under the direction of J. F. Poland, research geologist.

As one phase of the overall investigation, I am studying the physical and mineral characteristics of the water-bearing sediments that are compacting, the conditions under which the sediments were deposited, and the processes involved in their compaction. As background for the specific studies of these compacting sediments, this paper presents a review of what is currently known on the removal of water and the rearrangement of particles during the compaction of clayey sediments.

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FORCES RELATED TO CLAY-MINERAL SURFACES

Because of the large surface areas of clay minerals, the response of water-saturated clayey sediments to compacting pressures must be thought of in terms of the forces associated with clay-mineral surfaces. In very fine grained clays, such as montmorillonites, in which the surface areas may be as great as 800 m² per g (square meters per gram), the forces related to the clay-particle surface are more pertinent to the understanding of compaction than are the gravitational forces associated with the particle mass. This section of the review is an introductory and largely theoretical discussion of the surface forces. The role of these forces in compaction processes is one of the main subjects of the subsequent sections of the review.

FORCES BETWEEN CLAY-MINERAL PARTICLES AND WATER

HYDRATION OF CLAYS

The affinity between water and clay is well known. Dry clay will adsorb water vapor from the atmosphere. It will soak up large quantities of liquid water, if available, and swell to many times its dry volume. The cause of this attraction seems to be the negative charge on the surfaces of clay-mineral particles that arises from imperfections and substitutions within the mineral structure. The charge attracts water in at least two ways: directly, by attracting the protons of the polar water molecules; and indirectly, by attracting cations, which in turn attract water molecules. The hydration properties of the cations are significant, as the intensity and extent of the clay-water attraction are different for different adsorbed cations.

Several series of experiments have shown fairly conclusively that the water closest to the surfaces of montmorillonite or vermiculite particles is adsorbed or desorbed in molecular-layer units (Barshad, 1949; Collis-George, 1955; Foster and others, 1955, p. 299-300; Mooney and others, 1952; Norrish, 1954; Rowland and others, 1956; Walker, 1956). That is, if one allows a limited amount of water or water vapor to equilibrate with an originally dry clay, the water will first cover all accessible clay surfaces in a layer one molecule thick (about 2.5 Å¹). A second molecular layer will be adsorbed only after all accessible surfaces have been covered with the first layer. The pattern is reversed during drying or compaction: the layer closest to the clay-mineral surface is desorbed only after the complete removal of the next closest layer. Although the formation or removal of the third, fourth, and subsequent water layers varies with the type of clay mineral and adsorbed cation, the stepwise pattern of hydration and dehydration generally holds true for a distance from the particle surfaces equivalent to several layers of water.

PROPERTIES OF ADSORBED WATER

The water adsorbed on the surfaces of clay minerals has properties that differ from those of ordinary liquid water because it exists in a force field. Although this difference has been known for many years (see Terzaghi, 1925, p. 746), and although hundreds of experiments have been run to define quantitatively such properties as the density and viscosity of adsorbed water, very little more can be stated with certainty. Measurements of the characteristics of adsorbed water necessarily must be made on clay-water mixtures, and the presumed influence of the solid clay particles on the measure-

¹ Å = angstrom unit = 10⁻⁸ cm.

ments is subtracted from the whole to leave what is presumed to be the effect of adsorbed water. Interpretation of the experimental data therefore must rest on theoretical or conjectural assumptions whose validity is uncertain. The significant experimental work on adsorbed water was reviewed thoroughly by Low (1961) and Martin (1962). Significantly, these two authors—both of whom are highly qualified to review the subject—reached different conclusions about the structural arrangement of water on clay-mineral surfaces.

Low favored the idea that adsorbed water has a high degree of structural order and rigidity, making it more resistant than ordinary water to normal and shear stresses. Martin, on the other hand, favored a two-dimensional-fluid model in which the water is essentially fluid (perhaps more so than ordinary water) in directions parallel to the mineral surface and is essentially solid in the direction normal to the mineral surface; such an arrangement would make the water more resistant to normal stress but perhaps less resistant to shear stress than is ordinary water.

FORCES BETWEEN ADJACENT CLAY-MINERAL PARTICLES

Another way of considering the behavior of clay-water mixtures is in terms of the forces that operate between adjacent clay particles. Interparticle forces have received increasing attention and study by soil scientists and engineers since 1948, when Verwey and Overbeek published a theoretical treatment of the forces that operate between particles in colloidal suspensions (see also Overbeek, 1952). Their theoretical concepts were introduced to soil mechanics by Jiménez Salas and Serratosa (1953), Bolt (1956), and Lambe (1958a, p. 4-12; 1958b, p. 8-11) and have been useful in understanding the differences in behavior that seem to arise from variations in the chemical and physical components of soils. Because Verwey and Overbeek's theory was devised for dilute colloidal suspensions, its application to compacted sediments is limited. No theory has yet been devised that accounts for all the phenomena observed during compaction, and the available theory provides models that are useful as long as one keeps in mind their limitations.

The following discussion is taken from the papers cited above, plus Michaels (1958) and Parry (1960, p. 3.1-3.26). See also the critical discussion by Leonards (1962, p. 85-107).

REPULSION BETWEEN PARTICLES: DOUBLE-LAYER FORCES

The attraction between clay particles and adsorbed exchangeable ions in a clay-water mixture is opposed by the tendency of the ions to diffuse and distribute

themselves evenly in water. The result is a diffuse cluster of ions about a clay particle. This system can be considered a diffuse electric double layer: one layer is formed by the negative charge on the surface of the particle, and the other layer is formed by the concentration of exchangeable cations near the particle surface. The entire double layer—comprising the clay particle, exchangeable ions, and water—is an electric field, and the water in the field is attracted by an induced electrical force.

The particles in a clayey sediment repel each other because the outer parts of the double layers associated with the particles have the same net electrical charge (usually positive). The range and effectiveness of the forces of repulsion between particles are controlled by the thickness of the double layers, which probably ranges between 50 and 300 Å in most natural clayey materials and which varies systematically with certain factors. For example, the thickness of the double layer can be decreased by increasing the concentration of electrolytes in the water that occupies the pore space between clay particles. Increasing the concentration of electrolytes in the pore water inhibits the tendency of the adsorbed ions to diffuse and allows them to be held more closely to the surfaces of the clay particles. This added proximity, in turn, decreases the thickness of the double layer and shortens the distances over which the repulsive forces between particles are effective. Less pressure is then needed to compact the clay. The thickness of the double layer can be decreased in other ways: by replacing the adsorbed cations with cations of higher valence, or by increasing pressure.

EXPLANATION IN TERMS OF OSMOSIS

A convenient, but oversimplified, way to imagine the interaction of double layers is in terms of osmotic pressure. Because a clay particle in water attracts and holds cations, it serves the same function as the semi-permeable membrane in the classic osmosis experiment. The concentration of ions on the surfaces of clay particles relative to the concentration of ions in the bulk of the clay-water system determines the magnitude and direction of the osmotic pressure. When an aggregate of clay particles having ions attached is wet by water in which the concentration of electrolytes is low, the osmotic pressure is great and forces water between the clay particles, and the aggregate swells (fig. 1A). If the concentration of electrolytes is high (fig. 1B)—the difference between the cation concentration at the clay-mineral surfaces and that in the bulk solution is less—the osmotic pressure therefore is less, and the clay has less tendency to swell. The osmotic or swelling pressure is the force that must be overcome to compact the clay.

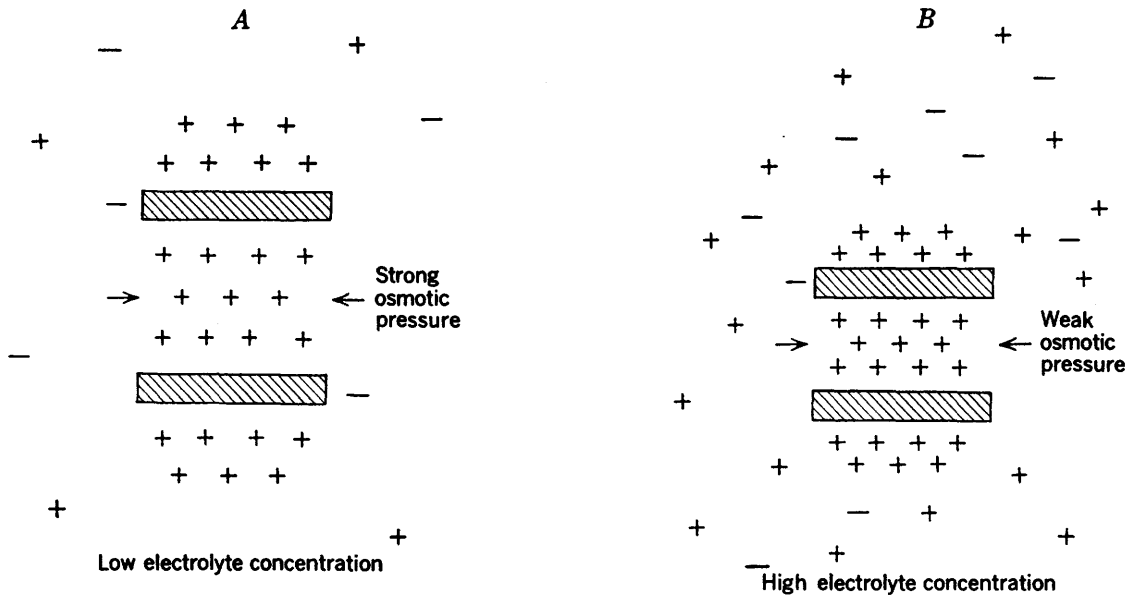


FIGURE 1.—Explanation, in terms of osmosis, of difference in swelling of clay aggregates in electrolyte solutions of (A) low and (B) high concentrations. Clay-mineral plates, viewed edgewise, represented by dark rectangles; cations and anions, represented by + and -, respectively.

Double-layer repulsion and the osmotic analogy, however, explain only part of the behavior of clay particles in water because they ignore the attractive forces that are effective when particles approach one another closely.

**ATTRACTION BETWEEN PARTICLES
VAN DER WAALS FORCES AND THEIR RELATION TO
DOUBLE-LAYER REPULSION**

Attractive van der Waals forces become significantly effective as the distance between clay-mineral particles is reduced to 10 or 20 Å. The intensity of these forces is an inverse function of a high power (fifth to sixth

power, probably, in most fine clays) of the interparticle distance. That is, the attractions are very strong at the particle surface and decrease rapidly in intensity away from the surface, as illustrated schematically by the dotted curve in figure 2A. Repulsive double-layer forces, represented by the dashed curve in figure 2A, are less intense at the particle surface, but their decrease with increasing distance between particles is less rapid. The resultant of the two types of forces (solid curve, fig. 2A) is net repulsion when particles are some distance apart and net attraction when they are very close together—on the order of 5 to 10 Å.

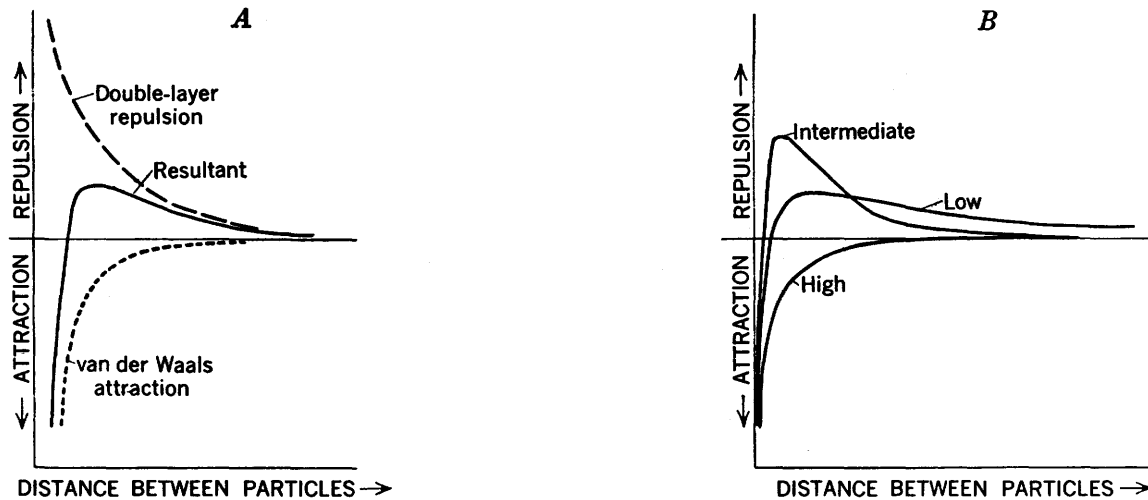


FIGURE 2.—Forces between basal (001) surfaces of adjacent clay-mineral particles, as a function of interparticle distance. Modified after Overbeek (1952, p. 272). A, Combination of repulsive double-layer and attractive van der Waals forces; B, effects of low, intermediate, and high concentration of electrolyte on net force between particles.

The electrolyte concentration, as discussed above, influences the thickness of the double layer. If the electrolyte concentration is great enough, it may suppress the repulsive forces to a distance that lies within the distance range of net attraction and thus cause the clay particles to attract one another. This is shown schematically in figure 2B and is a currently favored explanation for the influence of electrolyte concentration on the flocculation of clay particles.

EDGE-TO-FACE ATTRACTION

The above conceptual framework for the interaction between double-layer and van der Waals forces, intellectually satisfying as it is, unfortunately cannot account for all the forces observed in clay-water-electrolyte mixtures, mainly because it assumes that all particles are arranged so that their basal surfaces are parallel and because it ignores the forces associated with the nonbasal edges of the particles. The existence of positive charges on the edges of clay-mineral particles is strongly supported by several lines of evidence (most of them enumerated by Lambe, 1958a, p. 8), the most convincing of which is the edge-to-face arrangement of particles seen in electron micrographs of clayey sediments (Rosenqvist, 1962, pl. 5). The positively charged edges apparently are attracted to the negatively charged basal surfaces.

FACTORS INFLUENCING THE REMOVAL OF WATER DURING THE EARLY STAGES OF COMPACTION

"The water content which exists within a clay mass at any given time represents a balance between the urge of the clay minerals to suck in water and the tendency of applied loads to squeeze out water" (Lambe and Whitman, 1959, p. 39). Among the significant factors that influence the tendency of clay to retain water under small overburden loads—0 to about 50 kg per cm² (kilograms per square centimeter)—are the size and spatial arrangement of the particles, the clay-mineral constituents and their adsorbed cations, and the type and concentration of the interstitial electrolyte solutions. These factors are singled out here, not necessarily because they are the most significant influences, but because the literature contains enough material about them to make discussion profitable. The influence of other factors that may be significant—organic material, for instance—is almost completely unknown.

The influence of particle arrangement on water retention when the clay is under pressure is treated in more detail in the later sections of this review.

The present knowledge of the influence of the several factors, except for particle size, is based mainly on laboratory experiments. As nearly all these experiments

have been performed on selected size fractions of monomineralic clays in contact with distilled water or simple electrolytes, their results cannot be used to quantitatively predict the compaction of complex natural clay-water-electrolyte mixtures. In most experiments, furthermore, a single factor has been so isolated for study that one cannot tell whether the factor might be significant or inconsequential when it operates in concert with other factors in nature. The experiments, however, do indicate what the effects of some of the factors might be, and they provide questions that might be asked in any study of the early stages of natural compaction.

PARTICLE SIZE

The water content and compaction rate of fine-grained sediments are inverse functions of particle size. Figure 3A shows the decrease in void ratio (a measure of water content in saturated sediments) with increasing particle size in unconsolidated sea-bottom and lake sediments. Part of the variation in the void ratio of these groups is related to their different depths of burial, which are listed below.

	<i>Feet below sediment-water interface</i>
I. Lake Mead.....	0
II. Lake Maracaibo.....	0-18
III. Reservoirs in United States.....	0-10(?)
IV. Atlantic Ocean.....	0-9
V. Pacific and Arctic Oceans.....	0-7(?)

Most of the sediments in group III were dried at least once between the time they were deposited and the time they were sampled; the sediments in the other groups were never dewatered in their natural state. Figure 3B, which Skempton (1953, p. 55) derived from many different sediments in the United States and in Great Britain, shows that the relation between void ratio and particle size persists and that the fine sediments are compacted more rapidly than the coarser sediments under moderate overburden loads. The influence of particle size is also well demonstrated in the different void ratios and compaction rates of the different size fractions of illite shown in figure 8.

CLAY MINERALS

The difference in the void ratios of the three principal clay minerals under a wide range of pressures is shown in figure 4. Because the void ratio of these clays is a measure of their water content, the figure shows that under any given pressure in the range from 1 to 10,000 kg per cm², montmorillonite retains more water than illite, and illite, in turn, retains more water than kaolinite. The differences in water content are related principally to differences in particle size or, more precisely,

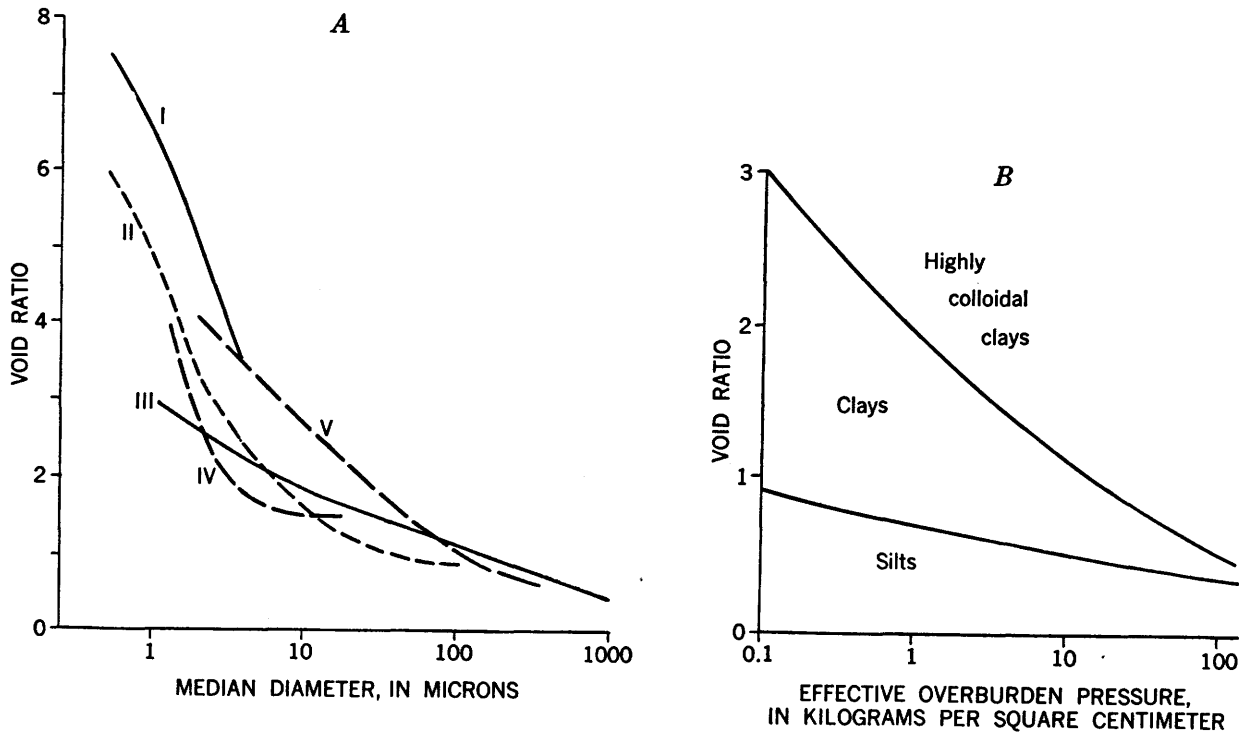


FIGURE 3.—Influence of particle size on void ratio of fine-grained sediments. A, Unconsolidated sediments. I, Lake Mead (Sherman, 1953, p. 399); II, Lake Maracaibo (Sarmiento and Kirby, 1962, p. 719); III, Reservoirs in Wyoming and other States (Hembree and others, 1952, p. 39); IV, Atlantic Ocean deep-sea deposits (Richards, 1962, p. 16); V, Pacific and Arctic Oceans, shallow and deep-sea deposits (data from Shumway, 1960, p. 454-463, and Moore, 1962, table 1). B, Generalized relation between size, void ratio, and effective overburden pressure. After Skempton (1953, p. 55).

to differences in specific surface (surface area per unit mass). The following data, from Bower and Goertzen (1959), Diamond and Kinter (1958), and Kinter and Diamond (1960), show the range of the specific surface of pure clay minerals:

	Specific surface (m^2 per g)
Montmorillonite	600-800
Illite	65-100
Kaolinite	5-30

The effect of specific surface on water sorption is shown schematically by Lambe's conception of typical sodium-saturated clay-mineral particles isolated in electrolyte-free water (fig. 5): a fairly large particle of kaolinite and a much smaller particle of montmorillonite, each adsorbing a water envelope of the same thickness. The montmorillonite particle thus adsorbs much more water relative to its mass than does the kaolinite particle.

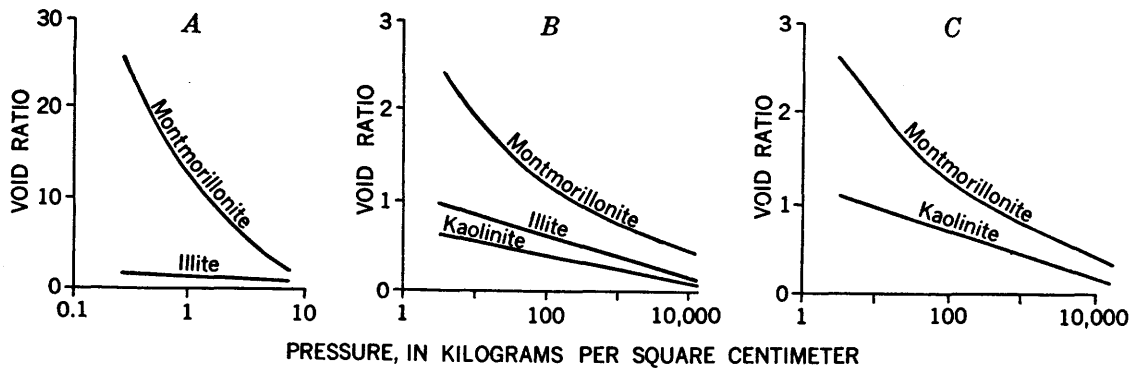


FIGURE 4.—Influence of clay minerals on relation between void ratio and compacting pressure. Note different void-ratio and pressure scales in A. A, At low pressure, saturated with $10^{-3} N$ NaCl. After Mitchell (1960, figs. M-3, M-7). B, At moderate to high pressure, saturated with distilled water. After Chilingar and Knight (1960, p. 104); modified by conversion of moisture contents to void ratios (assuming specific gravities: illite, 2.75 g per cm^3 ; montmorillonite and kaolinite, 2.6 g per cm^3) and by conversion of psi to kg per cm^2 . C, At moderate to high pressure, saturated with water (ionic composition not reported). After Kriukov and Komarova (1954); modified by conversion of moisture contents as in B.

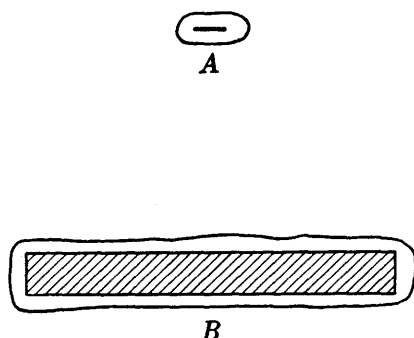


FIGURE 5.—Clay particles and their envelopes of adsorbed water. After Lambe (1961, p. 559). *A*, Typical montmorillonite particle, 1,000 by 10 Å; *B*, typical kaolinite particle, 10,000 by 1,000 Å.

Differences in chemical composition within the major mineral types may have a secondary influence on the water contents of clay minerals under pressure. Foster (1953, 1955) found a negative correlation between free swelling and the substitution of iron and magnesium for aluminum in the octahedral positions of 12 different montmorillonite samples. The exchange positions of each montmorillonite sample were saturated with sodium to standardize the effects of adsorbed ions. The swelling correlated much better with octahedral substitution than with either tetrahedral substitution or cation-exchange capacity. The differences do not seem to be due to charge deficiencies brought about by the substitution, because magnesium and trivalent iron caused about the same reduction in swelling volume. Rather the deficiencies seem to be due to changes in internal energy and bonding relations related to the differences in polarizing power between iron and magnesium on the one hand and aluminum on the other. Although Foster measured only the free swelling of the clays and not their behavior under increasing pressure, the different degrees of swelling should reflect at least qualitatively the degrees of resistance that the clays might offer to compaction.

INTERSTITIAL ELECTROLYTE SOLUTIONS

The interstitial electrolyte solutions seem to affect the response of clayey sediments to overburden loads in the range between 0 and about 10 kg per cm². The effect is a complex function of the type and concentration of electrolyte and of the size of the clay particles. In recent years, attempts have been made to rationalize these complexities in terms of the diffuse double-layer theory, and the theory seems to correspond fairly closely to the experimental observations made on mixtures of NaCl solutions and montmorillonite. Attempts at using the theory to predict the compaction and swelling behavior of other clay-water-electrolyte mixtures, however, have been only partly successful or entirely unsuccessful.

NaCl-MONTMORILLONITE-WATER MIXTURES

Because it provides an experimental verification of the diffuse double-layer theory, the NaCl-montmorillonite system probably has been given more attention than other electrolyte-clay mixtures. Experiments by Mitchell (1960), for example, showed some of the effects that the theory predicts (fig. 6): the greater interparticle distances (larger void ratios) at a given pressure are associated with the smaller electrolyte concentrations, and the presence of nonclay particles dilutes but does not otherwise influence the response of montmorillonite to pressure (note in fig. 6*B* that the void-ratio scale is different from that in fig. 6*A*). Although the agreement between the theoretical and experimental results of Mitchell's study of unfractionated montmorillonite is not particularly close, other experiments on very fine grained montmorillonite (<0.2 micron) showed rather close agreement. (See fig. 7*A*; see also Bolt, 1956, p. 91, and Warkentin and Schofield, 1960, 1962.)

The agreement between observed and theoretical results suggests that NaCl-montmorillonite systems satisfy the assumptions made in applying the double-layer theory to the compaction of clays. Some of these assumptions are worth examining because they do not seem to be satisfied by other electrolyte-clay systems. In addition to the validity of the Gouy-Chapman diffuse double-layer theory and the van't Hoff osmotic equation, which are used to compute the thickness of the double layer for a given pressure (see Bolt, 1956, p. 89-90), one must assume a model of the clay-water system in which the basal planes of all particles are parallel to one another and normal to the compacting stress and in which the distance between particles is uniform for any given pressure and electrolyte concentration.

Differences in particle orientation probably account for the different degrees of agreement between observed and theoretical compaction behavior in the first and subsequent compressions of montmorillonite reported by Warkentin and others (1957) (fig. 7*A*). During the first compression (open circles, fig. 7*A*), the observed interparticle spacings were greater than those predicted from theory (dashed line), probably because the orientation of montmorillonite flakes was not parallel. The compression, however, apparently improved the orientation, and the subsequent decompressions and recompressions yielded values very close to those predicted from theory. Had Mitchell (1960) repeated his loading and unloading several times, perhaps his measured values would have eventually coincided with his theoretical curves (fig. 6).

That sodium montmorillonites satisfy the assumption of uniform interparticle distance for a given electrolyte

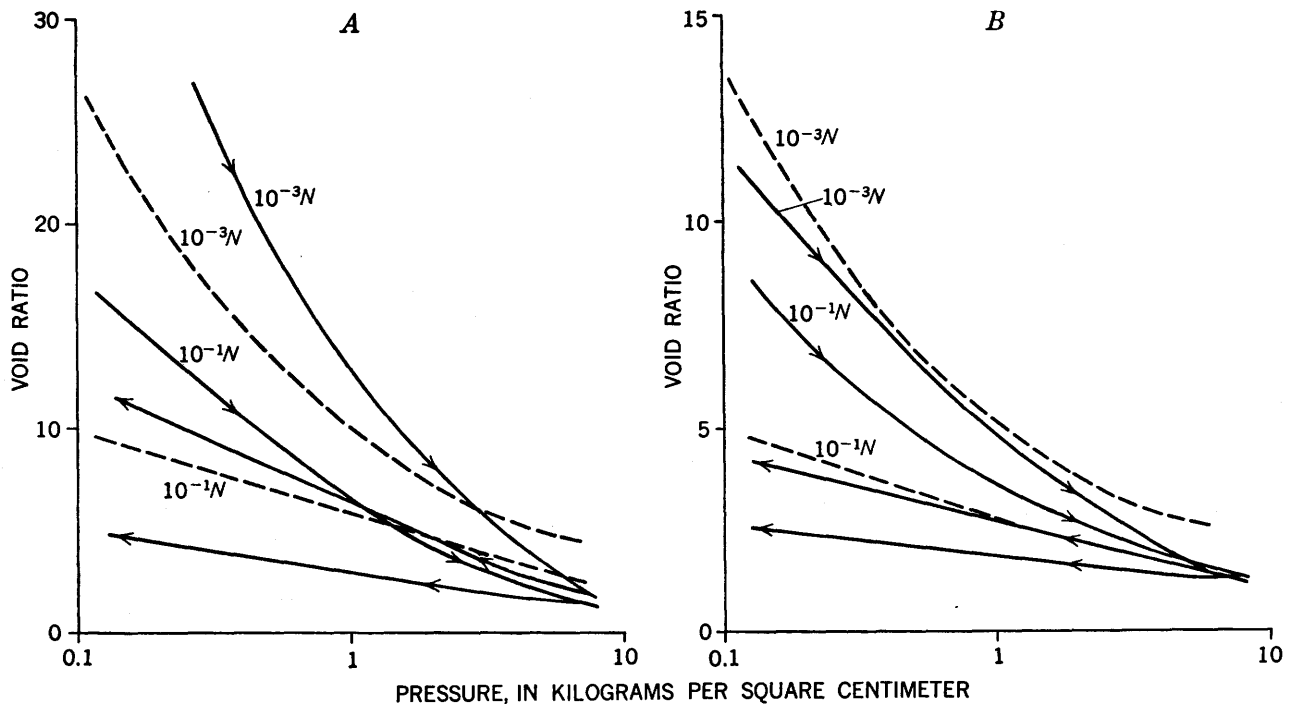


FIGURE 6.—Influence of NaCl concentration on relation between void ratio and pressure in montmorillonite. After Mitchell (1960, figs. M-7, M-8). Solid lines represent experimental results; dashed lines represent theoretical curves. A, Montmorillonite alone; B, montmorillonite plus silt (50 percent each by weight).

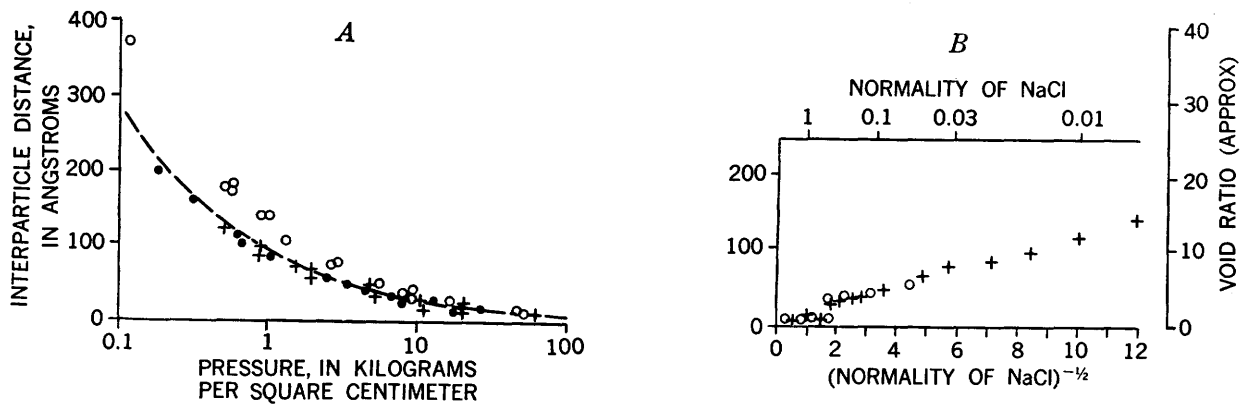


FIGURE 7.—Relations between NaCl concentration, interparticle spacing, and pressure in montmorillonite. A, Experimental and theoretical relations between interparticle spacing and pressure in a mixture of <0.2-micron fraction of montmorillonite and 10^{-4} N NaCl. After Warkentin and others (1957, p. 496). Open circles indicate first compression; solid circles, first decompression. Pluses indicate second compression, second decompression, and third compression. Dashed line is theoretical curve. B, Measured relations between normality of NaCl solution and interparticle spacing in dilute suspensions of <0.1-micron fractions of montmorillonite. Data indicated by open circles from Foster and others (1955, p. 302), by pluses from Norrish (1954, p. 124); farthest right measurement by Norrish reported by Quirk (1960, p. 2.2).

concentration was suggested by the experiments of Foster and others (1955) and of Norrish (1954; also described by Norrish and Quirk, 1954), which are shown in figure 7B. Both these experiments showed an inverse linear relation between interparticle distance (measured by X-ray diffraction) and the square root of NaCl concentration at water contents greater than 50 percent by weight (void ratio about 2.6) and at electrolyte concentrations less than 0.3 N. At lesser water contents or at greater electrolyte concentrations, the interparticle distance diminishes rather abruptly to 20A or less, which

corresponds in thickness to one, two, or three molecular layers of water. At water contents greater than 50 percent, the diffuse double layer apparently has the opportunity to form fully; at lesser water contents, the double layer does not seem to form, and the double-layer theory no longer accounts for the thickness of the water layers adsorbed on the particle surfaces. Graphs showing interparticle distance plotted against water content in sodium montmorillonites, not reproduced here, are similar to figure 7B. (See also Foster and others, 1955, p. 300; Hight and others, 1962, p. 506-509;

and either Norrish, 1954, p. 126, or Norrish and Quirk, 1954, p. 257.)

NaCl-ILLITE-WATER MIXTURES

The influence of NaCl concentration on the compaction behavior of illite varies with particle size. This was shown in the results of experiments on the type illite from Fithian, Ill. by Bolt (1956, p. 92) and by Mitchell (1960) (fig. 8). In very fine grained illite (<0.2 micron, fig. 8A), as in the NaCl-montmorillonite system, the greater void ratio at a given pressure is associated with the more dilute electrolyte. Illite particles of this size are apparently so small that their behavior is controlled mostly by surface forces that can be predicted qualitatively (but not quantitatively—note the difference between the theoretical and measured curves) from double-layer theory. The opposite relation is found in the coarser illite (<1.0 micron, fig. 8B): the greater void ratios are associated with the more concentrated electrolyte. This effect is even more pronounced in the coarsest illite (fig. 8C).

The coarser illite particles are apparently large enough to be affected somewhat by gravitational forces related to their mass, and their platy shapes make them susceptible to rearrangement under pressure. Only a part of their compaction takes place by decreasing the thicknesses of their double layers; the rest of it comes about through mechanical rearrangement. More pressure is required to rearrange the coarser particles that are in equilibrium with a more concentrated electrolyte because at higher concentrations of electrolytes the particles are more likely to flocculate. Once flocculated,

they are not as free to rearrange as they would be if they had been subject only to the repulsive interaction of double layers; therefore, they offer more resistance to compaction.

One compression experiment on a <2-micron fraction of kaolinite mixed with 1 *N* and 10⁻³ *N* solutions of NaCl (Mitchell, 1960, fig. M-4) showed the same relation between void ratio and NaCl concentration as that observed in the coarser grained illites.

OTHER ELECTROLYTE-MONTMORILLONITE-WATER MIXTURES

Clays mixed with other electrolytes do not always respond to compacting pressures in the same way as do clays mixed with NaCl. Montmorillonites that are mixed with electrolyte solutions containing calcium, magnesium, or aluminum, and whose exchange positions are saturated with these cations, will not swell to interparticle distances greater than 9 Å, regardless of the dilution of the interstitial electrolyte (Norrish, 1954; also reported by Norrish and Quirk, 1954). Apparently, some minimum-energy situation (perhaps an appropriate balance of repulsive double-layer and attractive van der Waals forces) exists at this spacing, beyond which the montmorillonite crystal will not expand freely. Likewise, montmorillonites saturated with potassium do not seem to expand readily beyond 5 or 6 Å with decreasing KCl concentration (Foster and others, 1955, p. 302-303; Norrish, 1954, p. 124). This implies that the water in these clays occupies two different types of pore space: interlamellar spaces, whose thickness does not exceed 9 Å, within the clay aggregates; and "external" pore spaces between the clay aggregates. The existence of two distinctly different sizes of pore space in calcium montmorillonite is supported by the nitrogen-sorption measurements reported by Aylmore and Quirk (1962, p. 109-112). The compaction behavior of these clays, therefore, cannot be completely understood on the basis of double-layer theory because one of the main assumptions—uniform interparticle distance at a given electrolyte concentration—is not satisfied.

An example was provided by the experiment of Blackmore and Miller (1961), the results of which are shown in figure 9. They compressed a mixture of montmorillonite and 2 × 10⁻⁴ *N* CaCl₂ solution under selected pressures and allowed it to reswell after release of pressure. The initial compressions followed fairly closely the pattern predicted from double-layer theory (dashed line). The reswelling, however, did not follow the theoretical pattern, thus suggesting that the montmorillonite flakes were compressed into aggregates that remained intact after the pressure was released. X-ray measurements supported the idea that these ag-

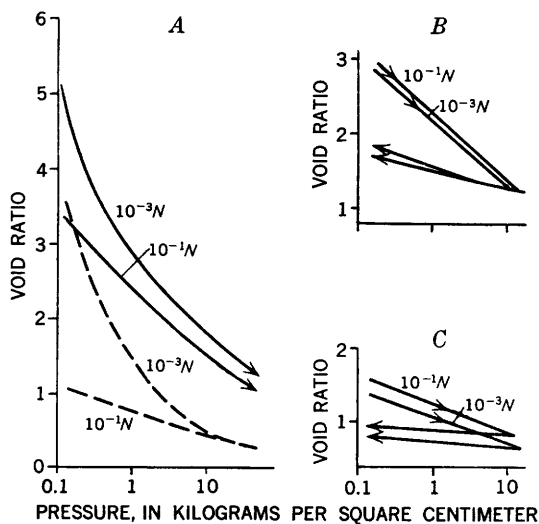


FIGURE 8.—Influence of NaCl concentration and particle size on relation between void ratio and pressure in Fithian illite. Solid lines represent experimental results; dashed lines represent theoretical curves. A, <0.2-micron fraction. After Bolt (1956, p. 92; also reported by Bolt and Miller, 1955, p. 287). B, <1.0-micron fraction. After Mitchell (1960, fig. M-5). C, Unfractionated. After Mitchell (1960, fig. M-3).

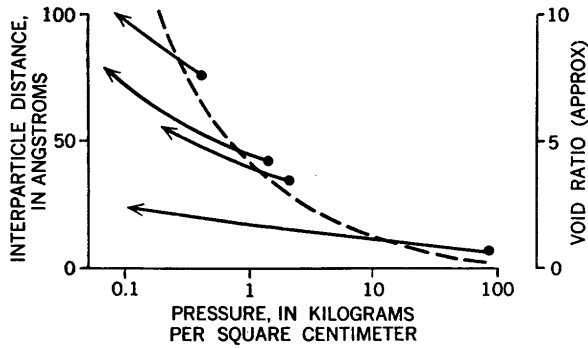


FIGURE 9.—Experimental and theoretical relations between interparticle spacing and pressure in mixture of montmorillonite and $2 \times 10^{-4} N$ CaCl_2 solution. After Blackmore and Miller (1961, p. 170). Theoretical curve indicated by dashed line.

gregates were “domains”—packets of several parallel unit montmorillonite sheets—within which the interlamellar distance remained a constant 8.8 Å, regardless of the total water content of the clay.

MgCl_2 (OR CaCl_2)-ILLITE-WATER MIXTURES

Mixtures of <2.0-micron fractions of Fithian illite and solutions of MgCl_2 and CaCl_2 were studied by Olson and Mitronovas (1962). The results of their compression studies of illite mixed with different solutions of MgCl_2 are shown in figure 10: these results apply also to CaCl_2 solutions, as the effects of calcium seem to be very similar to those of magnesium. The experiments showed the greatest resistance to compression at MgCl_2 concentration near $10^{-2} N$. The other four curves in figure 10A show a decrease in void ratio

with decreasing concentration. The effects of different electrolyte concentrations seem to disappear at the maximum pressure of about 30 kg per cm^2 . The rebound curves for all concentrations are essentially identical.

The observed decrease in void ratio with decreasing electrolyte concentration is contrary to the predictions of double-layer theory (compare fig. 10A, B). This reversal corresponds to the behavior of coarse-grained illite in NaCl solutions (fig. 8B, C) and can be explained in terms of the same model of particle rearrangement and flocculation. Olson and Mitronovas (1962, p. 193–194) did not believe that the influence of electrolyte content on the void ratios of these illites was especially significant: they found that they could produce the same degree of variation in void ratio by varying other factors such as particle arrangement and initial water content.

All these observations on the influence of electrolytes and electrolyte concentration were made on simple systems—mixtures of monomineralic clays with solutions of a single electrolyte, mixtures in which the cation of the electrolyte was the same as the cation adsorbed on the clay-mineral surfaces. In them one can find only suggestions of what might be the influence of multiple-electrolyte solutions on multiminerallc clays or of what one might expect in mixtures in which one cation predominates in the pore solution and another cation predominates in the assemblage adsorbed on the particle surfaces.

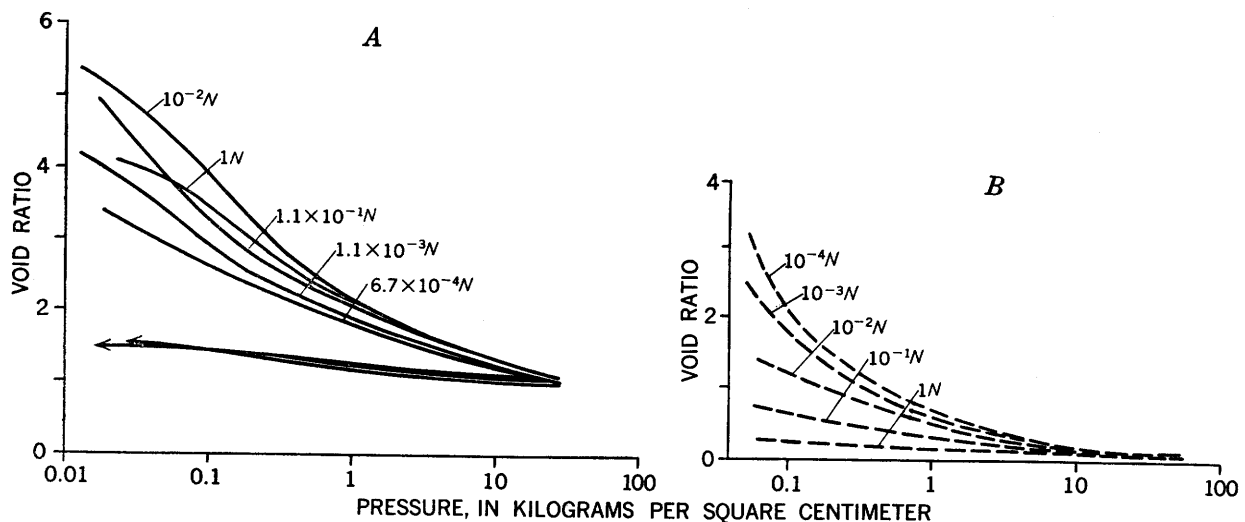


FIGURE 10.—Influence of MgCl_2 concentration on relation between void ratio and pressure in <2.0-micron fractions of illite. After Olson and Mitronovas (1962, p. 195, 197). A, Experimental; B, predicted from double-layer theory.

EXCHANGEABLE CATIONS

Another approach to understanding the chemical influences on clay compaction is to compare the behavior of clays whose exchange positions are saturated with different ions. Most of the experimental work done so far has tested the effects of different inorganic cations. Very little is known of the influence of adsorbed anions on the relation between water and pressure in clays. Likewise, except for a few compression experiments on clays mixed with organic liquids (mostly alcohols—see Jiménez Salas and Serratos, 1953, p. 196–198; Waidelich, 1958), very little is known of the effects of organic ions and molecules.

Because of its large exchange capacity, montmorillonite has been used in most of the studies of the influence of different cations. Results of three studies of some of the common cations are shown in figure 11. Montmorillonite saturated with sodium has a consistently larger void ratio at a given pressure than does montmorillonite saturated with aluminum, potassium, or calcium (or, presumably, magnesium, whose influence seems to be about the same as that of calcium). This difference is apparently related to the ability of sodium montmorillonite crystals to swell freely (fig. 7*B*), and to the limited ability of the other montmorillonites to swell to interparticle distances greater than 9Å. The difference in the void ratios at low pressures between Bolt's experiments (fig. 11*A*) and the other two experiments (fig. 11*B*, *C*) is related to differences in experimental conditions—Bolt began using a greater initial water content and used a finer grained montmorillonite.

Thomas and Moody (1962, p. 154) also measured the water contents of montmorillonite saturated with sodium, calcium, and aluminum at pressures of 0.33 and 15 kg per cm²: their results, which are too brief to be included in the figure, are similar to those shown in figure 11*C*.

Although Samuels' work (fig. 11*C*) suggested that differences in void ratio arising from differences in the exchangeable cations are insignificant at pressures greater than 10 kg per cm², Bolt's results (fig. 11*A*, representing very fine grained montmorillonite) suggested that these differences may be significant at somewhat greater pressures. The inference from Bolt's results is supported by a study of montmorillonite-rich sediments in the San Joaquin Valley of California (Meade, 1963), which suggests that the influence of different exchangeable cations may be significant in natural sediments at pressures as great as 50 kg per cm².

Other experiments by Samuels showed that the influence of exchangeable cations on the void ratio of kaolinite are different from their influence on the void ratio of montmorillonite. Results of his experiments on fractionated kaolinite (80 percent finer than 1 micron, 98 percent finer than 2 microns) are given in figure 12. Comparison with figure 11 shows two differences: the influence of different cations on the void ratio of kaolinite is much less than their influence on the void ratio of montmorillonite, and the general relation between void ratio and cation valence is reversed. Because clays flocculate more readily in solutions that contain cations having

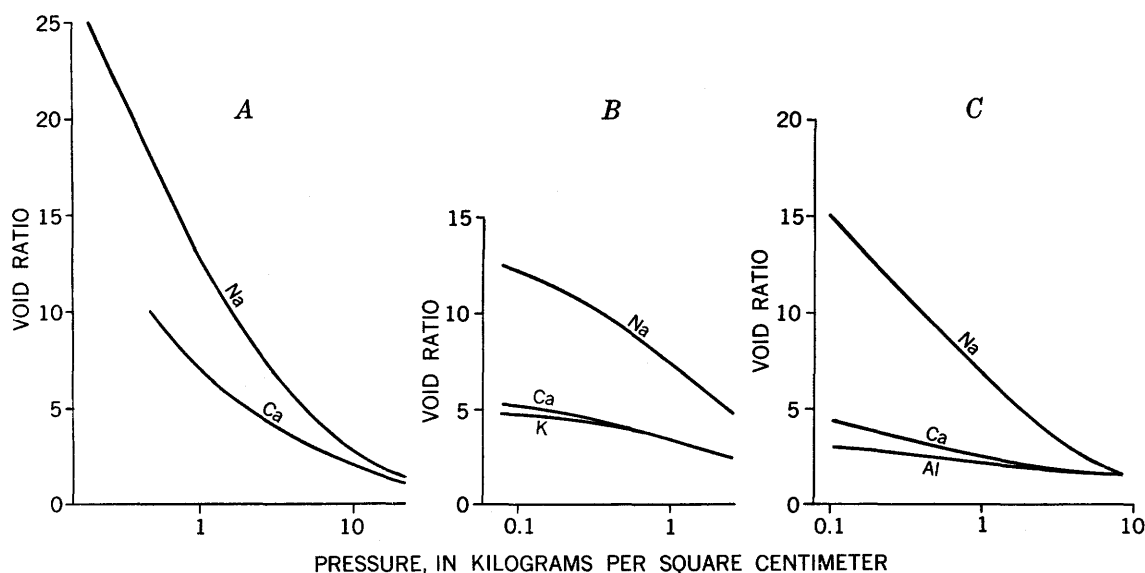


FIGURE 11.—Influence of exchangeable cations on relation between void ratio and pressure in montmorillonite. *A*, 0.2μ fraction mixed with 10^{-3} molar chloride solutions of indicated cations. After Bolt (1956, p. 91). *B*, 1.1μ fraction mixed with hydroxide solutions (concentrations unspecified) of indicated cations. After Jiménez Salas and Serratos (1953, p. 194). *C*, Presumably unfractionated and mixed with distilled water. After Samuels (1950; cited and described by Grim, 1962, p. 255–256).

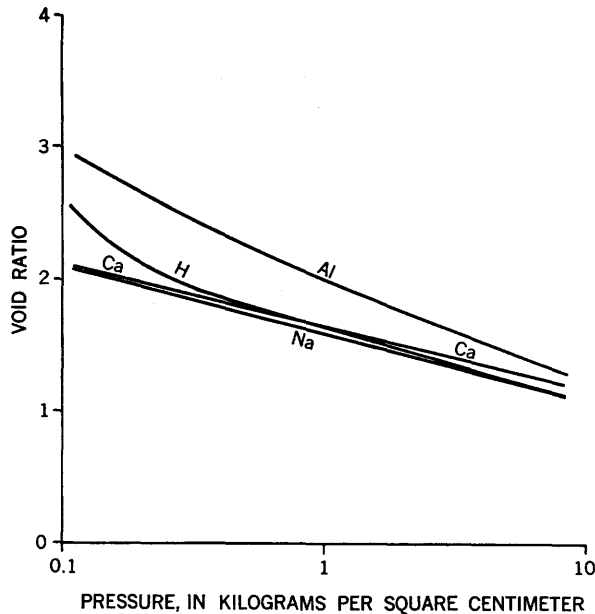


FIGURE 12.—Influence of exchangeable cations on relation between void ratio and pressure in kaolinite. After Samuels (1950; data also reported by Grim, 1962, p. 257).

high valences, perhaps this reversal in kaolinite represents the effects of particle flocculation that were invoked above to explain the relation between void ratio and electrolyte concentration in coarse-grained illite.

REMOVAL OF WATER DURING THE LATER STAGES OF COMPACTION

Compared to the removal of water during the early stages of compaction, which has been studied intensively in recent years by soil scientists and foundation engineers, the removal of water during the later stages of compaction has received little specific attention. Some general observations can be made, however, mainly on the basis of experiments conducted by von Engelhardt and Gaida (1963) and by Van Olphen (1963).

Of the factors that seem to influence the amount of water held in clayey sediments under low pressures, the clay-mineral composition and particle size also seem to be effective at higher pressures. Apparently, the amount of surface area available for water sorption is a major governing factor over the entire range of pressures to which natural clayey sediments are subjected. Electrolyte concentration and exchangeable cations, on the other hand, do not seem to influence the relation between water content and pressures greater than about 30–50 kg per cm². Experiments by von Engelhardt and Gaida (1963, p. 924–925) showed essentially no difference in the equilibrium void ratios, at any given pressure in the range from 30 to 3,100 kg per cm², either of montmorillonite mixed with NaCl solutions, CaCl₂ solutions, and distilled water, or of mixtures of montmoril-

lonite and NaCl solutions varying in concentration between 4.6 *N* and pure water. The only effect noticed was an increase in the rate of compaction—that is, the rate of water expulsion—with increasing NaCl concentration. Apparently the physicochemical influences of the different cation types and electrolyte concentrations do not affect the amount of water held by a clay unless the amount exceeds a certain minimum necessary to form diffuse double layers around the particles. This minimum amount seems to be about 50 percent by weight in very fine-grained (<0.1 micron) sodium montmorillonite; it should be somewhat less in coarser grained and more silty clays. When overburden loads have reduced the amount of water in a clayey sediment below this minimum, the forces that must be overcome to compact the sediment further are more conveniently thought of as forces of hydration—the attractions between the clay surfaces and water or between cations and water—rather than as forces of repulsion or attraction between particles.

The energies involved in the later stages of water expulsion have been estimated from measurements of the temperatures and negative water-vapor pressures required to remove the last few molecular layers of water from clays. From published data on the desorption of montmorillonites in response to decreasing water-vapor pressure, Van Olphen (1963, p. 182) estimated that the work required to remove the second and last layers of adsorbed water from calcium-saturated montmorillonite surfaces is equivalent to compacting pressures on the order of 2400 and 5200 kg per cm², respectively. These estimates are only approximate, but they indicate the enormity of the pressures required to squeeze the last increments of water from clayey sediments. Furthermore, these estimates are minimum values because they assume a parallel orientation of montmorillonite particles normal to the compacting stress—that is, they assume that the full load is borne by the water and that no load is borne by the solid particles. In nature, however, the particles probably will not lie in completely parallel positions but will touch one another at points of contact through which some of the load may be supported.

Adsorbed water can be removed from clay surfaces at temperatures that are fairly low in contrast to the large pressures required. Figure 13 shows pressure-temperature curves for the loss of the last layer of adsorbed water from montmorillonite surfaces at low water-vapor pressures as determined by Crowley and Roy (1959). Although the temperatures necessary to dehydrate the montmorillonites vary with the composition of the coordinated mineral lattice and with the exchangeable cations, they change only slightly with in-

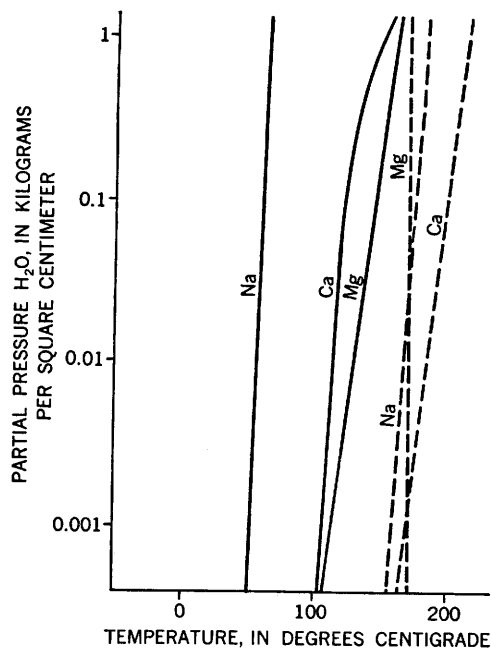


FIGURE 13.—Pressure-temperature curves for dehydration reactions of montmorillonite minerals saturated with sodium, calcium, and magnesium. After Crowley and Roy (1959, p. 19). Montmorillonite represented by solid lines; saponite by dashed lines.

creasing water-vapor pressure. If one considers the range of temperatures and pressures to which most natural sediments are subjected, temperature might be as significant a factor as pressure in removing the last increments of water from clays.

The combined influences of pressure and temperature were assessed by Van Olphen (1963, p. 183–186) in experiments on the removal of the last two molecular layers of water from a vermiculite clay. From the desorption of the vermiculite with decreasing water-vapor pressure at 25°C, he estimated (and his estimates were partly verified by X-ray measurements made during compression of the vermiculite to 2,000 kg per cm²) that the work involved in removing the second and last layers of water was equivalent to compacting pressures of 1,200 and 5,000 kg per cm², respectively. From a desorption experiment at a higher temperature, 50°C, he estimated the same two compacting pressures at 640 and 4,300 kg per cm². That is, an increase in temperature caused a noticeable decrease in the amount of pressure energy required to dehydrate the clay.

The removal of water during the late stages of the compaction of clayey sediments needs much more study—not only in terms of pressure and temperature, but also taking into account and assessing the significance of such factors as particle sizes, clay-mineral composition, and salinity.

FABRIC OF CLAYS AND ITS CHANGES UNDER PRESSURE

The discussion of the fabric of clayey sediments is in three steps. First discussed are some of the factors other than pressure that influence the arrangement of clay-mineral particles. Then follows a summary of the available information on the fabric of uncompacted natural sediments and, finally, a summary of experi-

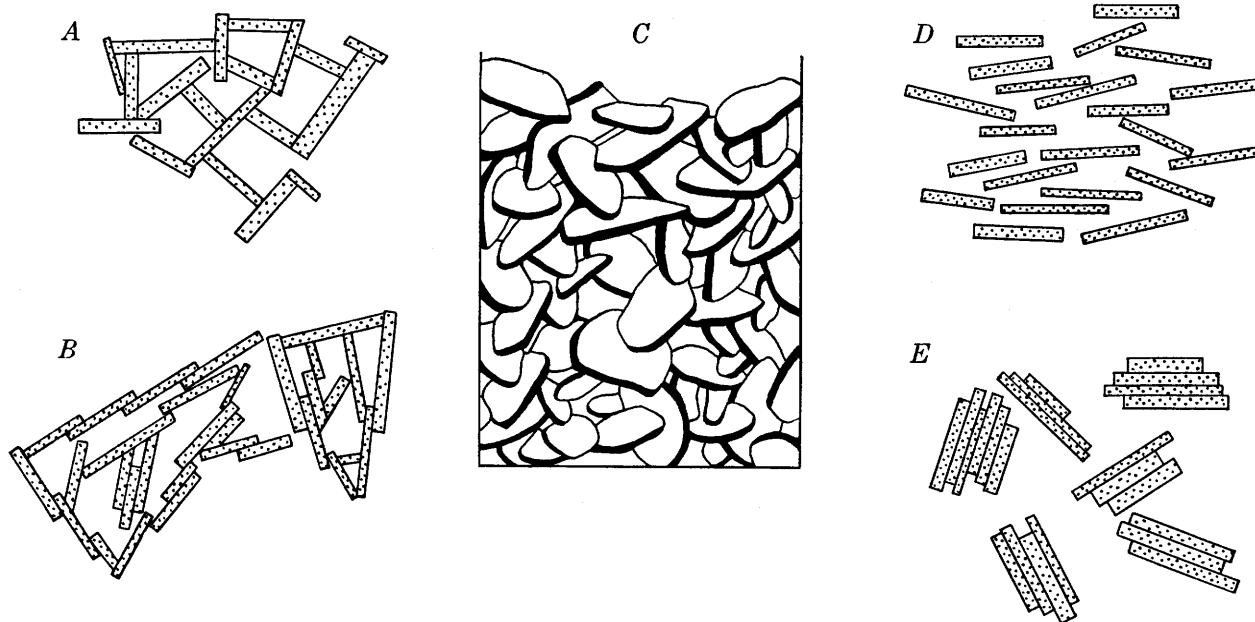


FIGURE 14.—Idealized arrangements of clay-mineral particles in sediments. A, Flocculated, edge to face, in salt-free water. After Lambe (1958a, p. 11). B, Flocculated, face to face, in salt solution. After Lambe (1958a, p. 11). C, Flocculated, edge to face. After Tan (1958, p. 87; 1959, p. 92). D, Preferentially oriented. E, Turbostratic.

mental and field observations on the influence of pressure on fabric.

Some of the more probable arrangements of platy clay-mineral particles in sediments are illustrated in figure 14. Schofield and Samson (1954, p. 142-143) and Lambe (1958a, p. 11) inferred the two types of flocculation shown in figure 14*A, B* from considerations of interparticle forces. In concentrated electrolyte solutions, the net force between particles may be attraction, resulting in a flocculated arrangement as shown in figure 14*B*. In electrolyte-deficient water, the principal attractive forces may be between the negatively charged basal surfaces and the positively charged particle edges, resulting in an edge-to-face arrangement as shown in figure 14*A*. A three-dimensional model of edge-to-face arrangement as visualized by Tan (1958, p. 87; 1959, p. 92) is shown in figure 14*C*. Preferred orientation of clay particles is represented in figure 14*D*. "Turbostratic" arrangement (name suggested by Aylmore and Quirk, 1960) is illustrated in figure 14*E*. It consists of domains—also called "tactoids," "packets," or "clusters"—of clay sheets within which the preferred orientation is nearly perfect and between which the orientation may be entirely random. The sequence *A-B-D* in figure 14 represents qualitatively a progression from random orientation to preferred orientation of particles.

FACTORS, OTHER THAN PRESSURE, THAT INFLUENCE THE ARRANGEMENT OF CLAY PARTICLES

Which of the factors that constitute a sedimentary environment might influence the arrangement of particles in a clayey sediment? On the basis of work published through 1962, the following chemical factors seem to be significant: electrolyte concentration, types of associated cations and anions, acidity or alkalinity, and associated organic matter. Mechanical factors of particle deposition also seem to be significant, but they have received less attention than have the chemical influences.

The available information on the relation between these factors and clay fabric is limited. Most of it is based on experiments in which the influence of individual factors has been isolated for detailed scrutiny; the combined influence of all factors in complex natural sediments is yet to be evaluated. Furthermore, most of these experiments recorded the influence of various factors at atmospheric pressure and only provided indirect suggestions of the influence that the factors might have in sediments under overburden loads. And, finally, the arrangements of particles in most of these experiments was inferred indirectly from observations of sedimentation volume and from the tendencies of the clays to aggregate or disperse in different chemical solutions.

In very few of these experiments has the fabric been measured directly by X-ray, optical, or other means.

ELECTROLYTE CONCENTRATION

Electrolyte concentration seems to have different effects on the fabrics of different clay minerals. It influences illite and kaolinite in one way, montmorillonite in another. Rosenqvist (1955, p. 53-62) experimented with the sedimentation of 10-percent suspensions of illite in NaCl solutions ranging in concentration between 0.002 and 0.5 *N* and found that the equilibrium volume of settled-out sediment increased with increasing concentration of NaCl. Similar results were obtained by Hsi and Clifton (1962, p. 272) in their experiments with 1- to 2-percent suspensions of kaolinite and metahalloysite that had flocculated in solutions of NaCl, CaCl₂, FeCl₂, and AlCl₃ ranging in concentration from 0.0003 to 0.02 *N*. If one assumes that the volume of the sediment reflects the degree of disorder of the particle arrangement (random orientation of the particles with regard to each other), these results suggest that the tendency toward random orientation of illite and kaolinite particles is a direct function of the electrolyte concentration. The arrangement of particles flocculated by the more concentrated electrolytes is presumed to be as shown in figure 14*B*.

In montmorillonite, on the other hand, the relation between sedimentation volume and electrolyte concentration seems to be either inverse or nonexistent, depending on the cation involved. Hofmann and Hausdorf (1945) experimented with 0.4- to 4.0-percent suspensions of montmorillonite in NaCl and KCl solutions ranging in concentration from 0.004 to 2.0 *N* and found that the equilibrium volume of the sediment that settled out decreased with increasing electrolyte concentration. In CaCl₂ and MgCl₂ solutions, the sedimentation volume and interparticle distance (9-10 Å) remained essentially constant at all concentrations between 0.004 *N* and 2.0 *N*. These observations are similar to those cited in the discussion of the influence of different electrolytes on the removal of water from montmorillonites under low pressures (figs. 6, 7, 9) and are perhaps best explained in the same terms—conformity or lack of conformity with the predictions of the double-layer theory.

The spatial arrangement of montmorillonite particles in dilute NaCl solutions—if it is in fact a function of the double-layer repulsion between particles—should be similar to the arrangement illustrated in figure 14*D*. That is, the particles are probably dispersed in the solution in an array that approaches parallel orientation, the interparticle distance varying with electrolyte concentration as shown in figure 7*B*. Because the sedimentation volume and interparticle distance of mont-

morillonite do not vary with the concentration of CaCl_2 and MgCl_2 , the arrangement of particles in these solutions probably is not a salt-flocculated arrangement but something similar to the collection of parallel aggregates illustrated in figure 14E.

These interpretations of the influence of electrolyte concentration on clay fabric are based on several assumptions—different assumptions, moreover, for montmorillonite and for the other clay minerals—and should be regarded as tentative. The spatial arrangement of clay minerals in different solutions needs to be studied by some means more direct than inference from the sedimentation volume.

CATIONS AND ANIONS

In the experiments of Hsi and Clifton (1962) on the flocculation of kaolinite and halloysite in solutions of NaCl , CaCl_2 , and AlCl_3 , the volumes of the settled-out suspensions in the different electrolytes increased in the order Na^+ , Ca^{+2} , Al^{+3} . This increase suggests that the higher valence cations are associated with a more random particle arrangement; the lower valence cations, with a greater degree of preferred orientation. The suggestion is supported by Samuels' observations on kaolinite under pressure (fig. 12) but is contradicted by the observed influence of cations on the void ratio of montmorillonite under pressure (fig. 11). Apparently, as is suggested by the experiments on electrolyte concentration, the different cations have a different influence on the different clay minerals.

Properties of the cation other than the valence also seem to influence the settling volume, as shown by experiments of Rosenqvist (1955, p. 59; 1959, p. 40) on illites sedimented in 0.75 *N* concentrations of NaCl and KCl . He reported that the volume of the sediment that settled in the KCl solution was about 30 percent greater than the volume that settled in the NaCl solution. Experiments with illites that settled in 0.75 *N* solutions of LiCl (smaller settling volume than in NaCl) and CsCl (greater settling volume than in KCl) indicate further that valence is not the only cation property that influences the arrangement of particles. Lambe (1958a, p. 7, 9) suggested, on the basis of double-layer considerations, that this influence on the settling volume is an inverse effect of the size of the hydrated ion, which seems to decrease in the order $\text{Li} > \text{Na} > \text{K} > \text{Cs}$. That is, the smaller hydrated ions allow the illite particles to approach one another more closely, to flocculate more readily, and to orient more randomly.

The influence of different anions on the arrangement of clay particles is essentially unknown. Hsi and Clifton (1962) studied the influence of NaCl , Na_2SO_4 , and Na_2CO_3 on the flocculation of kaolinite and halloysite, and Longenecker (1960, p. 188) measured the

settling volumes of a montmorillonite-rich clay in different concentrations of NaCl and Na_2SO_4 . Except for the observation that the montmorillonite-rich clay has a larger equilibrium sedimentation volume in Na_2SO_4 solutions than in NaCl solutions of equivalent normality, little more can be said than that the anions are likely to influence the fabric.

ACIDITY

Schofield and Samson (1953, 1954) found an apparent relation between acidity and flocculation in 10-percent suspensions of kaolinite. Purified sodium kaolinite flocculated spontaneously in distilled water but deflocculated on addition of small amounts of NaOH . Measurements of the adsorption and desorption of chloride ions by the kaolinite led Schofield and Samson to conclude (1) that the flocculation resulted from attractions between positively charged particle edges and negatively charged particle faces (fig. 14A), and (2) that the positive edge charges (and hence the tendency to flocculate) were strongest under acid conditions, diminished with increasing pH, and eventually became negative.

Sisler (1960, p. 192) measured the sedimentation volume of samples of mud from the bottom of Lake Mead after he allowed them to settle in HCl or NaOH solutions adjusted to a series of pH values between 2 and 12. Judging from Rolfe's (1957) analyses of sediments from the same part of Lake Mead (Boulder Basin), the mineral constituents of the mud are probably quartz, illite, and kaolinite, plus minor amounts of montmorillonite and feldspar. The largest sedimentation volume—1 percent greater than the volume at pH 7.2 (approximately the natural pH)—was observed at pH 2. The smallest volume—22 percent less than at pH 7.2—was observed at pH 10. These results suggest that the tendency for clays to assume an open flocculated arrangement is substantially less at strongly alkaline pH than at neutral or strongly acid pH.

ORGANIC MATTER

Organic matter probably influences the arrangement of clay particles in sediments. Studies by Bloomfield (1956), Lawson and Keilen (1951), and Söderblom (1960), among others, indicated that kaolinite or illite can be flocculated or deflocculated by solutions of some of the organic materials that are likely to be associated with sediments—aqueous extracts from tree leaves, bark, wood pulp, or peat—but these studies contain no direct indications of the arrangement of particles.

Ingram (1953) observed a direct correlation between the presence of organic material and the preferred orientation of clay-mineral particles in 50 clayey rocks from Colorado, Iowa, and Wisconsin. The rocks ranged in age from Ordovician to Eocene. Ingram

made his observations from thin sections of the rocks. He noted (p. 873-875) that organic stain was associated with clays that showed preferred orientation and that it was absent from clays in which the orientation was random. In one rock sample, this relation was expressed in alternate organic and nonorganic clayey layers. Organic matter also seemed to influence the fabric of the clay minerals when the rocks were disaggregated, suspended, and then sedimented from suspensions by adding NaCl. Clay minerals from rocks that contained organic material settled out of suspension in a pronounced parallel arrangement, whereas the arrangement of sedimented particles from the non-organic rocks was mostly random.

PHYSICAL CONDITIONS OF DEPOSITION

In addition to being influenced by chemical factors, the fabric of a clayey sediment must also be affected by physical and mechanical factors involved in deposition: rate of deposition, state of agitation or quiescence of the water in which the sediment is deposited, particle-size distribution and concentration of the sediment being deposited, and drying of the sediment between deposition and burial.

The concentration of the sediment in the depositing medium, for example, seems to influence the degree to which the particles assume whatever equilibrium fabric is favored by the existing chemical conditions. Raitburd (1960, p. 110) reported that, whereas kaolinite particles settled out of 2- to 5-percent suspensions to form a preferentially-oriented fabric (measured by X-ray diffraction), no predominant orientation of particles was observed in kaolinite that had settled out of a 50-percent (by weight?) suspension. In the experiments of Olson and Mitronovas (1962, p. 192), a calcium illite sedimented from a very dilute suspension maintained a larger void ratio under pressures in the range from 0 to 10 kg per cm² than did one sedimented from a 34-percent (by weight) suspension. This result suggests that the illite sedimented from the more dilute suspension assumed a more open flocculated arrangement, which influenced its response to pressure.

Results of experiments by Isaac Barshad (oral communication, 1963) suggested that drying may have irreversible effects on the fabric of some montmorillonites. In his experiments, well-dispersed montmorillonites were first saturated with sodium, potassium, calcium, or magnesium and sedimented by centrifuging. The montmorillonites then were dried, dispersed again mechanically, and sedimented once more by centrifuging under the same conditions as before. The amounts of water retained by the sedimented clays are expressed volumetrically in the following void ratios:

Exchangeable cation	Void ratio	
	After first centrifuging	After drying and recentrifuging
Sodium or potassium.....	~40	~40
Calcium or magnesium.....	18-25	6-8

These results suggested that drying irreversibly increases the size of the domains into which calcium and magnesium montmorillonites are aggregated—an effect similar to the irreversible compression of calcium montmorillonites that was observed by Blackmore and Miller (1961) and is represented in figure 9. The wet volume (and presumably the fabric) of sodium and potassium montmorillonites, on the other hand, does not seem to be influenced significantly by intermediate drying.

FABRIC OF UNCOMPACTED NATURAL CLAYS

Most of the specific information on the fabric of recently deposited and unconsolidated clays comes from the electron micrographs of illitic clays from Scandinavia made by Rosenqvist (1958, p. 441; 1959, p. 39; 1962, pl. 5). In the marine clays that he examined, Rosenqvist (1959, p. 38) found an openwork arrangement of particles dominated by contacts between corners and planes, corresponding “to an astonishing degree” with the arrangement as visualized by Tan (fig. 14C). Rosenqvist reported further (1962, p. 25) that, in about a hundred stereoscopic electron micrographs of unconsolidated and unweathered marine clays from Scandinavia, he had “* * * never observed anything resembling a domain structure.” In fresh-water clays, on the other hand, he has found a greater degree of parallel orientation between clay particles—in arrangements approaching that shown in figure 14D. Rosenqvist related the difference between the fabrics of marine and fresh-water clays to the flocculating effect of the electrolytes in sea water. In fresh water, the illite particles apparently did not aggregate readily but settled individually out of suspension into more efficiently packed arrangements.

Rosenqvist's observations on the different fabrics of marine and fresh-water clays are partly corroborated by observations made in other parts of the world in sediments buried under a few tens of feet of overburden. At these depths, presumably, the fabric reflects its original state more than it reflects the influence of pressure. Raitburd (1960, p. 113, 115) reported completely random orientation—measured by X-ray diffraction—of illite and kaolinite particles in a Quaternary marine clay from the northeastern part of the Black Sea. Mitchell (1956), from his thin-section study of 14 marine and nonmarine clays from North America, reported generally better formed preferred orientation in the nonmarine clays. Wu (1958), on the

other hand, found different degrees of random and preferred orientation in glacial-lake clays from the southern Great Lakes area that could not be related to different types or concentrations of ions in the interstitial waters.

An indirect indication of the conditions under which clays might be flocculated at fairly low salinities is given by evidence from studies of the sediments transported by the Colorado River and deposited in Lake Mead. Sherman (1953, p. 396-400) compared the settling velocities of fine-grained lake sediments in the lake water with those of the same sediments in solutions of a dispersing agent and found that many of the particles in nature were aggregated into floccules. Howard (1960, p. 104) found the same type of floccules suspended in the waters of the Colorado River at Grand Canyon, 145 river miles upstream from Lake Mead. Sherman also compared the pore volumes of natural lake sediments with those of sediments that had been dispersed artificially. The natural sediments having median diameters of about 1 micron had an average void ratio of about 6 (curve I, fig. 3A); sediment of the same size that had been dispersed with a chemical agent and allowed to settle out of suspension had a void ratio of about 2. Although these observations give no indication of the degree of orientation, they suggest that the arrangement of fine particles transported by the Colorado River and deposited on the bottom of Lake Mead might be less preferred than are the orientations observed in Scandinavian lakes by Rosenqvist. The clay minerals in Lake Mead and presumably in the river sediment are illite, montmorillonite, and kaolinite, in proportions that vary from one part of the lake to another (Rolfe, 1957). The pH of slurries of the lake sediment ranges from 7.0 to 7.6 (Rolfe, 1957, p. 380). The principal ions dissolved in the lake and river water are Na^+ , Ca^{+2} , SO_4^{+2} , and HCO_3^{-1} , and the total salinity usually ranges between about 300 and 1200 parts per million (Howard, 1960, p. 108). Expressed as normality, this concentration range is about 0.005 to 0.02 *N*, which was the range of concentrations of the sodium and calcium salts that was sufficient to flocculate kaolinite minerals in some, but not in all, of the experiments of Hsi and Clifton (1962).

In addition to most of the fabrics observed in clayey sediments, clayey soils exhibit some peculiar particle arrangements of their own. Oriented coatings on silt and sand grains, oriented films on the walls of cracks or pores, and domainlike aggregates seem to be formed in soils by elutriation, repetitive drying, and leaching processes. For details of these processes and fabrics, see Brewer (1956), Minashina (1959), and Rosenqvist (1962, p. 23-25).

INFLUENCE OF PRESSURE ON THE FABRIC OF CLAYS

The removal of water under the influence of pressure implies that the solid particles in a clayey sediment must move closer together into a more efficiently packed arrangement. At least two special arrangements—preferred orientation and turbostratic orientation (fig. 14D, E)—may be formed during compaction. These two fabrics, and particularly the preferred orientation, have received most of the emphasis in compaction studies. One should keep in mind, however, that other responses to pressure such as the bending and crumpling of clay particles are also possible.

FABRIC OF CLAYS COMPRESSED IN THE LABORATORY

Laboratory experiments on the rearrangement of particles in water-saturated clays under uniaxial pressure can be divided into two groups: those in which the sides of the clay are not confined but are allowed to spread freely, and those in which the sides of the clay are rigidly confined. Neither condition is an exact replication of the distribution of pressures during natural compaction, but the laterally confined experiments probably represent nature more closely.

UNCONFINED COMPRESSION

Preferred orientation is produced readily during unconfined compression. Aggregates of Georgia kaolinite were subjected to uniaxial compressions of 0.22 and 0.55 kg per cm^2 by Buessem and Nagy (1954). X-ray powder photographs showed that the orientation of kaolinite flakes in the uncompressed aggregates was random and that compression increased the orientation of the flakes normal to the direction of applied load. Williamson (1947) produced preferred orientation by repeatedly dropping a ball of illite-kaolinite clay onto a glass plate from a height of about 1 foot; he produced the same orientation by squeezing a similar ball of clay between parallel glass plates in a hand press. Raitburd (1958, p. 791) compressed a cylinder of kaolinite paste between glass plates to one-third its original thickness and produced a clearly detectable (by X-ray diffraction) preferred orientation. Popov (1944) observed preferred orientation in fluid montmorillonite pastes (water, 40-70 percent by weight) that he produced by squeezing the pastes between glass slides. The combination of compression and lateral flow apparently favors the formation of preferred orientation.

COMPRESSION OF LATERALLY CONFINED CLAYS

Some clays form preferred orientation under low pressure when they are laterally confined. Mitchell (1956) mixed an illite-quartz powder with sea water, placed it in a rigid-walled cylinder, and subjected it to a uniaxial load of 4 kg per cm^2 . A thin section of the compressed clay showed that the illite particles, initially

in random orientation, were in preferred orientation in the plane normal to the compressive stress. He also studied the effects of uniaxial pressures of 2 kg per cm² in a group of 14 marine and nonmarine clays from North America. In the marine clays, in which the predominant clay mineral was illite, the compression caused an increase in the degree of preferred orientation over large areas as well as within small domain-like aggregates (generally less than 0.5 mm in diameter). In the nonmarine clays, most of which contained substantial proportions of montmorillonite, the results of the compression were not consistent; the degree of preferred orientation was increased in some samples and decreased in others.

Domains also seem to form in some clays under low to moderate pressures. From measurements of the sharpness of X-ray reflections from compressed calcium montmorillonite, Blackmore and Miller (1961, p. 171) inferred that the number of montmorillonite unit 10-Å thick sheets per domain increased progressively with pressure, ranging from about 5 unit sheets at 0.5 kg per cm² to nearly 8 unit sheets at 90 kg per cm². The process seemed to be irreversible; that is, the sheets in the domains did not seem to dissociate when the pressure was released.

Preferred orientation seems to form readily under great pressures applied in laboratory experiments. Engelhardt and Gaida (1963, p. 925-926) observed an increase in the degree of preferred orientation of montmorillonite and kaolinite particles with increasing pressures between 80 and 800 kg per cm². Olson (1962, p. 34, 37) produced an extreme degree of preferred orientation by cyclically compressing and decompressing calcium illite 15 times at pressures between 4.4 and 4,500 kg per cm². Raitburd (1960, p. 111-113) made a series of uniaxial compression experiments on pastes of different clay minerals—presumably kaolinite and montmorillonite. He did not state the range of pressures used in these experiments, but one can infer from other experiments described in his paper that the pressures may have been as great as 5,000 or 10,000 kg per cm². When thin pastes (1.5 mm) were compressed, a homogeneous orientation formed normal to the compressive stress. When thicker pastes (2 cm) were compressed, orientation formed in two directions: one normal to the compression, and the other along slip planes at about 45° to the direction of compressive stress. The particle orientation in all these high-pressure experiments was measured by X-ray diffraction.

Evidence of bending and crumpling of fine-grained clay particles under pressure is scarce. Tan (1959, p. 93) stated that montmorillonite particles may be broken under heavy local stress concentrations, but he did not

cite evidence or give an idea of the magnitude of pressures he had in mind. Norton and Johnson (1944) said that kaolinite particles averaging 0.32 micron in diameter and 0.04 micron in thickness were distorted by pressures as low as 40 kg per cm²; but again no direct evidence was cited.

INFLUENCE OF ELECTROLYTE CONCENTRATION UNDER PRESSURE

Von Engelhardt and Gaida (1963, p. 926) used X-ray diffraction to measure the bulk degree of preferred orientation of montmorillonite compressed under 800 kg per cm² in three solutions of NaCl ranging in concentration from 0.16 to 1.1 *N*. They found an inverse relation between the concentration of NaCl and the degree of preferred orientation at right angles to the compacting pressure. They found also that, whereas the void ratio remained constant at about 0.5 in all three samples, the permeability (as measured by the rate of compaction) increased with increasing NaCl concentration. These observations can be explained by a greater degree of preferred particle arrangement (fig. 14*D*) at smaller concentrations and by a more turbostratic arrangement (fig. 14*E*) at larger concentrations. Figures 14*D* and 14*E* are drawn with approximately equal void ratios and show the effect of fabric on permeability—water moves more readily through large pore spaces.

In addition to providing the most satisfactory model to explain the above observations, a turbostratic fabric seems reasonable in a mixture of montmorillonite and 1.1 *N* NaCl when one recalls studies that were discussed earlier in this review. The experiments illustrated in figure 7*B* indicated that, at NaCl concentrations greater than about 0.3 *N*, montmorillonites do not expand readily to interparticle spacings greater than 9 Å. Montmorillonites mixed with calcium electrolytes do not expand readily beyond interparticle spacings of 9 Å at any concentration. Figure 9 and other observations suggest that particles of calcium-saturated montmorillonite under load are compressed irreversibly into domains. Perhaps montmorillonite in contact with concentrated NaCl solutions behaves in the same way.

FABRIC OF NATURALLY COMPACTED CLAYEY SEDIMENTS

Only a few detailed studies have been made of the orientation of clay-mineral particles in shales, and, with the exception of one study, there is little evidence to support the formation of oriented fabrics—either preferred or turbostratic—during natural compaction.

In a recent study of a 2,000-foot-thick section of unconsolidated nonmarine sediments (Meade, 1961*b*), X-ray diffraction measurements showed no progressive increase of the degree of orientation of montmorillonite

particles parallel to the bedding with increasing depth (fig. 15). The only significant preferred orientation was found in a fresh-water-lake deposit, which suggests that the fabric might not have been produced by compacting pressures but by the slow settling of individual clay particles in the still waters of the lake. In the rest of the sediments, mainly alluvial-fan and flood-plain deposits, no preferred orientation seems to have formed either during deposition or as a result of compaction.

If one considers that von Engelhardt and Gaida (1963) were able in the laboratory to produce an increase in the degree of preferred orientation of pure montmorillonite with increasing pressures between 80 and 800 kg per cm², the lack of preferred orientation in the alluvial sediments represented in figure 15 may be due to one or more of the following reasons:

1. Perhaps not enough pressure has been exerted on the natural sediments to cause any discernible preferred orientation in the montmorillonite; the maximum effective load to which these sediments have been subjected is 60 to 70 kg per cm².
2. Perhaps the presence of silt and sand particles inhibits the formation of preferred orientation in the natural sediment.
3. Perhaps the montmorillonites have been compressed into domains (which are not detectible by the X-ray method) rather than into a homogeneous parallel fabric.

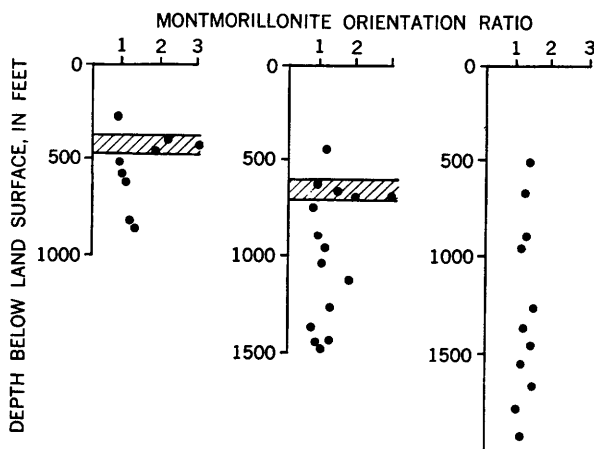


FIGURE 15.—Horizontal preferred orientation of montmorillonite in unconsolidated clayey sediments from three core holes in San Joaquin Valley, Calif. Montmorillonite orientation ratio computed from X-ray-diffraction peak heights (explained by Meade, 1961a): the larger the ratio, the greater the degree of preferred orientation parallel to the bedding. Ratio near 1.0 signifies random orientation. Lacustrine desposits cross-hatched.

The formation of domains is a likely possibility for two reasons: (1) The principal exchangeable cation is calcium, and (2) domainlike aggregates that were deposited as such (shale fragments and other aggregates) are available for nucleation of further material.

Kaarsberg (1959) found, in flat-lying shales buried under 2,700 feet or more of overburden in several parts of the United States and Canada, that the preferred orientation of illite increased with the depth of burial. Illite is the predominant clay mineral in all the natural sediments he studied. Many of them contain chlorite; only a few have even minor amounts of montmorillonite. He used X-ray diffraction and sound velocities to measure preferred orientation, and he used dry bulk density as a measure of compaction. A sample of his measurements is tabulated below. Using a method described elsewhere (Meade, 1961a), I computed the "orientation ratio" from X-ray diffraction measurements that Kaarsberg had plotted in a graph on page 469 of his report. The larger the ratio, the greater the degree of preferred orientation of illite particles parallel to the bedding. Other X-ray diffraction measurements plus sound velocities support Kaarsberg's conclusion (p. 470-471) " * * * that the degree of preferred orientation of the basal planes of the illite particles parallel to the bedding increases with compaction."

Results of Kaarsberg's study (1959)

[Orientation ratio computed by Meade]

Type of sample	Present depth below surface (ft)	Bulk density (g per cm ³)	Orientation ratio
Recent clay	0	1.9	1.5
Cretaceous shale	4,340	2.4	2.8
Do	5,136	2.5	2.2
Do	6,776	2.6	3.2
Do	9,120	2.7	5.0
Slate		2.9	32.0

In many older shales, however, increasing degree of preferred orientation with increasing depth of burial has not been observed. White (1961, p. 564), for example, stated that the fissility (which is presumably a reflection of fabric) of Paleozoic shales in Illinois does not vary systematically with the depth of burial. In the same group of Paleozoic shales, Grim and others (1957) noted a relation between preferred orientation of clay minerals and particle size; preferred orientation was better formed in fine-grained shales (in which most of the particles were smaller than 2 microns) than in coarser grained shales that contained appreciable amounts of nonclay mineral grains. The influence of natural compaction on the fabric of clayey sediments apparently needs to be evaluated in conjunction with other likely influential factors.

SUMMARY

The removal of water and the rearrangement of particles in clayey sediments under pressure are complex functions of particle size, clay minerals and associated ions, interstitial electrolyte concentration, acidity, temperature, and the arrangement of particles at the onset of compaction. Other factors such as associated organic material may influence the compaction processes, but very little is known about them.

Particle size is perhaps the most significant factor—not only in its consistently inverse relation to pore volume under a wide range of pressures, but also in the sense that it influences the nature and degree of the influence that the other factors have on the progress of compaction. In very fine grained clays, which have large areas of particle surface available to interact with interstitial water and dissolved ions, the influence of electrolytes and exchangeable ions can be expressed in terms of the forces of repulsion or attraction related to the particle surfaces. In coarser grained clays, on the other hand, the surface forces become less significant, and one must give more consideration to the rearrangement of particles in response to gravitational forces associated with the particle mass.

The different influences of cation type and electrolyte concentration under low overburden pressures seem to be as follows:

1. In fine-grained clays (montmorillonites and fine-grained illite):
 - (a) In sodium solutions less concentrated than about 0.3 *N*, the water content at equilibrium (and perhaps the degree of preferred orientation of the particles?) increases with decreasing concentration.
 - (b) In sodium solutions more concentrated than about 0.3 *N* or in potassium, calcium, magnesium, or aluminum electrolyte solutions, the variation of water content with electrolyte concentration is less apparent because of the tendency of the clays to aggregate irreversibly into oriented domains in which the interparticle distance does not exceed 9 Å. This tendency is reinforced by overburden pressure during compaction.
2. In coarse-grained clays (kaolinite and coarse-grained illite), the water content at equilibrium and the randomness of particle orientation increase with increasing electrolyte concentration or with increasing cation valence. This relationship apparently results because the coarse clays flocculate more readily in the more concentrated solutions or

in higher valence cation solutions to form open-work structures that resist the compacting effects of small overburden pressures.

These observations, however, were made on single-cation, single-electrolyte, single-mineral systems and have only suggested what might be observed in more complex systems.

The response of clayey sediments to compacting pressures greater than about 50 kg per cm² seems to be influenced mainly by the particle size, clay minerals (whose influence is related mainly to their size), and temperature. Under these pressures the cation type and electrolyte concentration may influence the clay fabric, but they do not seem to influence the total amount of interstitial water held in the sediments. The main effect of increasing temperature is to reduce the amount of pressure necessary to remove the interstitial water.

Although preferred and turbostratic orientations can be produced by compressing clays in the laboratory, there is little evidence to show that these fabrics are formed readily and generally during natural compaction. Mainly on the basis of experimental evidence, the following factors might seem to encourage the formation of preferred orientation under overburden loads:

1. Existence of a partly oriented fabric at the onset of compaction. If preferred orientation is already incipient in the sediment, it would seem more likely to form to a greater degree during compaction than if the particles were in some other kind of arrangement.
2. Increasing size of clay-mineral particles. Kaolinite and illite seem to be oriented more readily under pressure than is montmorillonite.
3. Decreasing concentration of interstitial electrolyte. This decrease should reduce the tendency of the coarser clays to flocculate and the tendency of the finer clays to aggregate into domains.
4. Decreasing cation valence also should reduce the tendency of the clays to flocculate or to form domains.
5. Decreasing acidity.
6. Presence of organic material.

The formation under pressure of a turbostratic fabric instead of a general degree of preferred orientation might be favored by increasing electrolyte concentration and increasing cation valence.

The influence of all these factors on compaction processes needs further study. Especially needed are studies of the combined influences of the factors and their relative significance under a wide variety of natural conditions.

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