THE OCCURRENCE OF SEPIOLITE AND ATTAPULGITE ON THE SOUTHERN HIGH PLAINS*

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Abstract — Sepiolite and attapulgite have been found to be common, sometimes the major, clay minerals in calcareous lacustrine deposits on the southern High Plains in West Texas and eastern New Mexico. Deflation debris derived from the basins and calcareous soils developed in the debris and in the lacustrine deposits also often contain either or both minerals. Dolomite is the carbonate commonly associated with sepiolite and calcite has a similar relationship to attapulgite in the lacustrine deposits. Pedogenic formation of sepiolite and attapulgite appears unlikely in the area studied since an association with lacustrine materials was made in a very high percentage of the occurrences.

Sepiolite was found to be highly concentrated in the $< 0.2\mu$ fraction. A similar, but less pronounced, distribution was noted for attapulgite. The studies suggest that the minerals have developed authigenically in alkaline lacustrine environments during periods of desiccation. Such an environment, interrupted by more humid periods, would have obtained during dry Pleistocene intervals. Volcanic ash is suggested as the source of the essential silica. The Mg concentration would appear to determine whether sepiolite–dolomite or attapulgite–calcite were formed.

INTRODUCTION

THE OLDEST Cenozoic deposits on the southern High Plains of Texas and New Mexico, the Ogallala Group, were formed by deposition of sandy alluvium from the northern and central New Mexico mountains in late Pliocene time. The often-indurated upper part of the group is sometimes referred to as "caprock caliche" (Evans and Meade, 1944).

Three localized Pleistocene formations (Blanco, Tule, and Tahoka) overlie the Ogallala Group. The Blanco Formation (Nebraskan age) crops out in Blanco Canyon, Crosby County and along Rita Blanca Creek in Hartley County. Sediments in both deposits range in texture from sand to clay and contain calcite, dolomite, and volcanic ash (Evans and Meade, 1944; Frye and Leonard, 1957).

The Tule Formation (Kansan age) crops out along Tule Creek in Briscoe and Swisher Counties and along Spring Creek in Garza County. The

variable-textured formation includes volcanic ash, bentonitic clay, and dolomite (Evans and Meade, 1944; Frye and Leonard, 1957).

The Tahoka Formation (Wisconsin age) is exposed along the western margins in several of the large saline playas in West Texas. These deposits are clay-rich, calcareous, and gypsiferous (Evans and Meade, 1944). Dune ridges resulting from deflation of the playas generally are extensive on the eastward margins.

In addition to the aforementioned clay-rich, lacustrine deposits, an Illinoian eolian mantle, informally called "Cover sands" by Frye and Leonard (1957), occurs extensively in the area. The "Cover sands" thicken from the northeast to the southwest and are the parent material for most of the mature soils of the area.

Prior to the initiation of the current study, sepiolite and attapulgite had been reported from only two sites in the area. Reeves and Parry (1968 a,b), in a study of the clays of several of the large saline playas, found both sepiolite and attapulgite in Mound Lake, a playa of approximately three square miles, in Lynn and Terry Counties, Texas (Fig. 1). Kunze (G. W. Kunze, Texas A & M University, unpublished mimeographed material) detected attapulgite in the calcareous substratum of a soil from Scurry County. In an area considerably removed to the southwest, Vanden Heuvel (1966) reported both sepiolite and attapulgite from
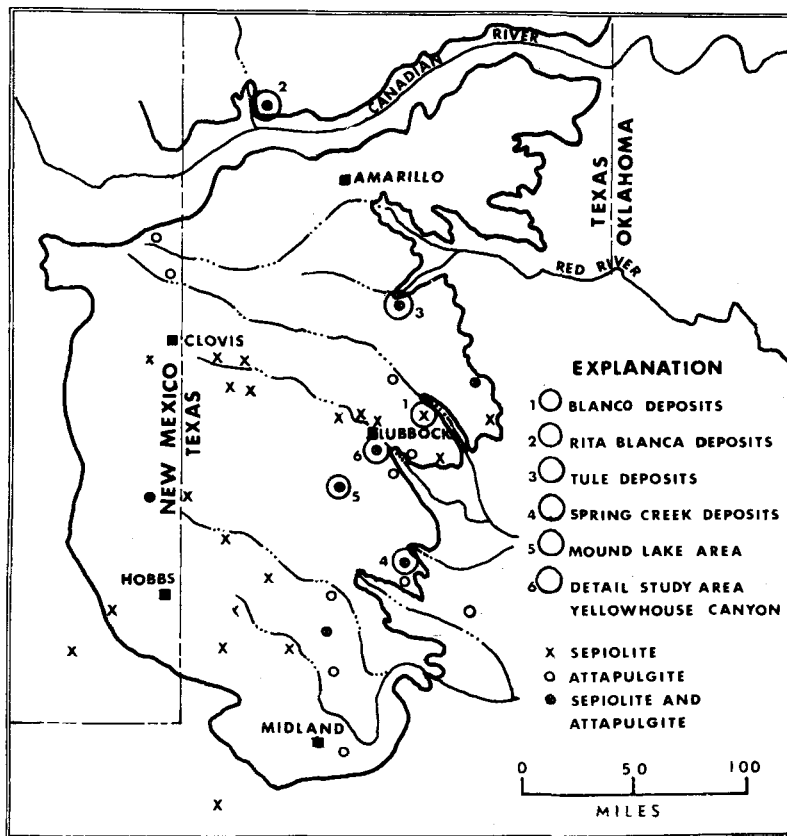
Fig. 1. Sampling sites on the Southern High Plains in West Texas and eastern New Mexico. Large circles numbered 1–4 indicate areas of major reported lacustrine deposits. Areas studied in detail in this investigation are numbered 5 and 6.

soil caliche near Las Cruces, New Mexico. He proposed a pedogenic origin for both minerals.

The study reported herein was undertaken initially to determine the mode of origin of the Mound Lake fibrous clays. Parry and Reeves (1968a) had proposed a lacustrine origin. However, because of the two known occurrences in soil caliche, it was thought that soils surrounding the playa could be the source of the minerals. It was found early in the study that several soils in the vicinity of the playa did indeed contain sepiolite, yet only calcareous soils derived from eolian materials located on the lee side of the playa or in those developed in outcropping Tahoka sediments contained the mineral. No trace of either sepiolite or attapulgite was detected in either the upper non-calcareous portion or in caliche substrata of soils derived from other parent materials in the vicinity of the playa. In a concurrent separate study, Leach (J. W. Leach, Texas Tech University, unpublished M.S. thesis) found sepiolite to be

abundant in core samples from the Mound Lake basin, outside the modern playa, as deep as 25 ft in the Tahoka Formation. Lesser amounts of attapulgite occurred in some units. The sepiolite that occurs in the Holocene lacustrine and eolian sediments is believed to have been derived from older sediments.

MATERIALS AND METHODS

Field sampling

To further study the question of lacustrine versus pedogenic origin the investigation was expanded to include samples from selected strata in the known Nebraskan (Blanco and Rita Blanca beds) and Kansan (Tule and Spring Creek beds) deposits of presumed lacustrine origin (Evans and Meade, 1944). Locations are shown in Fig. 1. Frye and Leonard (1957) proposed an alluvial origin for the same beds.

During the course of the study it was discovered that apparent lacustrine deposits are exposed for

many miles along Yellowhouse Canyon (called Yellowhouse Draw in the upper reaches) that passes through the City of Lubbock. Although Frye and Leonard (1957) reported Blanco-like deposits in the canyon southeast of Lubbock, the nature of the deposits had not been previously recognized. In fact, some of the exposures have been erroneously referred to as soil caliches. A section exposed in a small side tributary of the canyon was selected for detailed study (site 6, Fig. 1). Because of the discovery of these lacustrine deposits, samples were also taken from similar-appearing deposits along several other draws in the region. Also, several samples were selected from soil substrata in what were suspected to be ancient basins. Sampling was also done on a number of pedogenic caliches. Sampling sites are shown in Fig. 1.

Criteria used to distinguish lacustrine deposits were, (1) gray and bluish-gray sediments, indicative of reducing conditions, (2) quiet water sediments, such as dolomite, (3) evaporites, such as gypsum, and (4) geomorphic evidence of ancient basins with their adjacent deflation dunes. In nearly all cases at least three criteria could be applied. Most of the sepiolite- or attapulgite-containing strata were white because of the large amount of carbonate present. However, these were often interbedded with bluish-gray, less calcareous strata. Further, upon removal of the carbonates, the samples which contained the minerals were usually bluish-gray. In the area studied, gypsiferous soils seem to occur only in association with either modern or ancient basins.

Pedogenic caliches were distinguished by, (1) an enriched carbonate substratum (the soil "ca" zone) in association with non-calcareous, or in a few cases, much less calcareous, overlying soil horizons, (2) morphological features such as a laminated upper layer, and (3) light red and reddish-brown colors, indicative of oxidizing conditions. Reddish medium-textured material characteristic of the High Plains "Cover sands" was present below the "ca" zone. Again, because of the extremely high amount of carbonate present, the caliche was almost white but the reddish browns of the original sediment became apparent upon carbonate destruction. The caliches varied from weakly cemented and crumbly to strongly indurated.

Sample preparation

Considerable time was spent in the preliminary stages of the investigation in studying the effects of various pretreatments on sepiolite-containing samples from Mound Lake. Vanden Heuvel (1966) reported that when samples were dialyzed to re-move carbonates in buffered NaOAc (pH 5) for more than one hour considerable destruction of sepiolite occurred. We found that one hour was not long enough to remove sufficient carbonates to effectively study the clay mineralogy, even on samples that had been screened through a 300 mesh sieve. Further, we found that sharp, well-defined sepiolite peaks on X-ray diffractometer tracings could still be obtained on samples that had been dialyzed for several weeks in the buffered NaOAc. Subsequently, samples on which detailed mineralogical studies were planned were dialyzed until free of carbonates and then desalted in deionized water until dispersion within the dialysis tubes occurred; this is basically the procedure of Grossman and Millet (1961). Gypsiferous samples were treated in the same manner until the carbonates were destroyed. They were then dialyzed in tap water until free of gypsum and subsequently desalted in deionized water. We also found that the carbonates could be effectively removed with 5% HOAc without pH control and without apparent adverse effects on the sepiolite peaks. This method of carbonate removal was used in the remainder of the study when only a gross identification of the clay minerals was desired.

Samples on which a more detailed investigation was carried out were further dispersed by adding 10 ml of 5% Calgon solution and then agitated on a reciprocating shaker for several hr. The clay was then separated by repeated siphoning at the calculated time for a particle with an effective diameter of 2μ to settle. Volume control of the clay suspension was maintained by flocculation with 12% $CaCl_2$ solution. After clay separation was complete, the flocculated suspensions were again dialyzed until free of Cl^-. The clay samples were then dispersed with 5% Calgon solution before separation into coarse $(2-0\cdot2\mu)$ and fine $(< 0\cdot2\mu)$ fractions by centrifugation. A small portion of clay suspension was Ca saturated and glycerol solvated. Another small portion was saturated with potassium and first heated at 250°C for 4 hr, X-rayed, and then again heated for 4 hr at 550°C.

For clay samples used for gross mineral identification, dispersion was accomplished by repeated decantation of the supernatant liquid after the carbonates were destroyed. A few drops of the suspension were pipetted onto glass slides after sufficient time had been allowed for the sand and silt to settle. The slides were then air dired and stored over $CaCl_2$ until X-rayed.

X-ray diffraction

Nickel-filtered CuK_α radiation from a Norelco diffractometer equipped with a 1/2° divergence and scatter slits, along with a 0·006 in. receiving slit,

was used on all samples. The samples were scanned at 1° per min.

We found early in the study that the 12 Å (110) peak was diagnostic for sepiolite. The peak, present in the Ca–glycerated and K-250 diffractograms, disappeared with the 550° heat treatment (Fig. 2). The diffuse asymmetrical band at 10–11 Å that remains after the 550°C treatment may be caused by small quantities of illite but is also due to restructured sepiolite (Caillere and Henin, 1961). We also determined that the 10·5 Å (110) peak of attapulgite was most useful for identification. The 6·4 (200) line was not a reliable indicator unless the sample was exceedingly high in attapulgite. Attapulgite, when occurring in a mixed assemblage with illite, could be readily detected because of the pronounced diminution of the 10–10·5 Å peak area when heated to 550°C relative to the area obtained from the sample when heated to 250°C (Fig. 3). This method, i.e. relative change in the intensity of the 10–10·5 Å band between the 250°C and 550°C heatings, was subsequently used to identify attapulgite. Caillere and Henin (1961) have described a structural change in attapulgite at 400°C. The third-order illite peak at 3·3 Å was unaffected by the 550°C heating.

Other studies

Transmission electron micrographs for the study were made by John L. Brown, Consulting Physicist, Atlanta, Georgia. They were used early in the investigation to establish the dominance of an
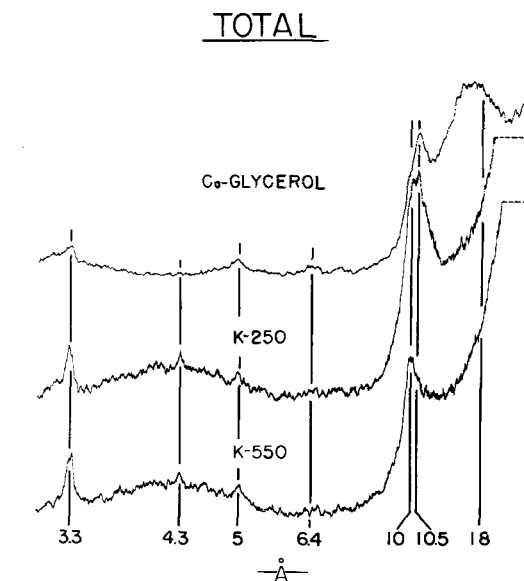

Fig. 3. X-ray diffractograms of total ($< 2\mu$) clay from an attapulgite-rich sample.

acicular mineral in a sample that gave a 12 Å peak, but not a 10·5 Å, peak. The same was done on a sample that gave a very strong 10·5 Å, but not a 12 Å, reflection. We believe this to be confirmation of the validity of the diffraction peaks used to identify the minerals. A micrograph of the 12 Å mineral is shown in Fig. 4. Several micrographs of

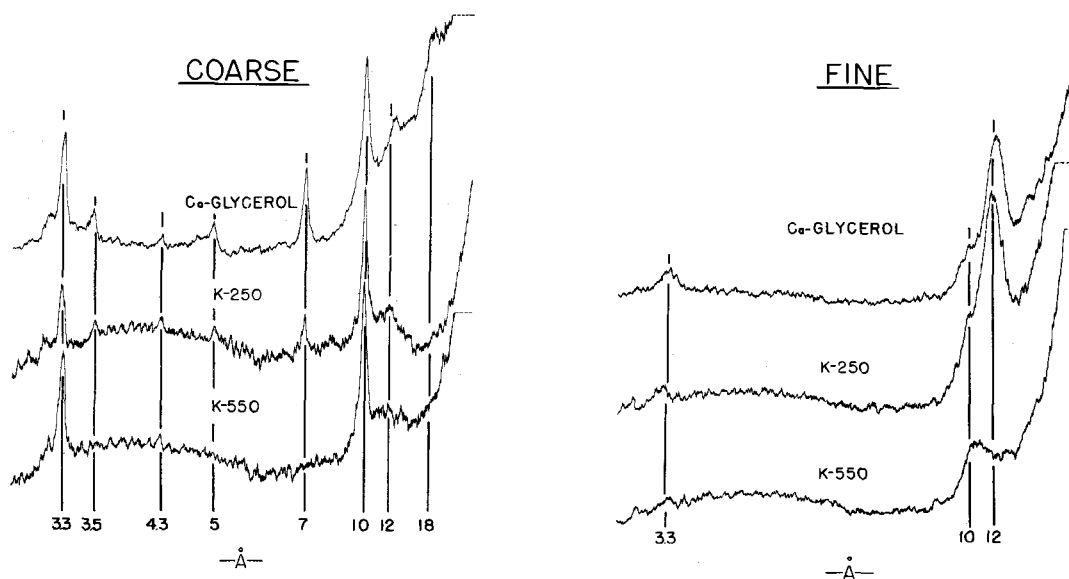Fig. 2. X-ray diffractograms of coarse (2–$0·2\mu$) and fine ($< 0·2\mu$) clay from a sepiolite-rich sample.

Fig. 4. Electron micrograph of fine clay for which the X-ray diffractogram is shown in Fig. 2.

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the 10·5 Å mineral, which showed a mineral with a morphology indistinguishable from the 12 Å mineral, were obtained, but none are shown here.

Total chemical analyses were performed by Coors Spectrochemical Laboratory, Golden, Colorado. Cation exchange capacity (CEC) measurements were made according to the method described in the USDA Handbook 60 (1954).

RESULTS AND DISCUSSION

Sample analyses showed that sepiolite and attapulgite are relatively common on the southern High Plains (Fig. 1). We detected either or both minerals in approx. 75% of the samples studied. Sepiolite was considerably more common than attapulgite and both minerals occurred in only a few samples. In all cases, except two, the minerals occurred in reported (Evans and Meade, 1944) or suspected lacustrine materials. Either or both acicular minerals occurred in selected strata from all four of the major reported Pleistocene lacustrine deposits. We observed that the white strata in such deposits which contained very high amounts of dolomite (as much as 75% $CaCO_3$ equivalent) usually also contained the purest sepiolite.

In a separate, more detailed study of the Blanco Formation, Rogers (D. M. Rogers, Texas Tech University, unpublished data) found that several strata contained sepiolite or attapulgite almost to the exclusion of any other clay mineral.

In one of the two occurrences which could not be related to apparent lacustrine deposits, sepiolite was detected in a caliche overlain by a soil near Hobbs, New Mexico. In the other, attapulgite was detected in a ravine exposure in the same general vicinity of the attapulgite found by Kunze (unpublished data). More work will be necessary in both areas before the origin of the carbonate material can be determined. We want to re-emphasize, however, that sepiolite and attapulgite occur in soil caliches, as well as in the sola (A and B horizons) of some calcareous soils of the region, but with the exception noted above, the soil parent materials are either lacustrine or eolian debris derived therefrom.

A general mineralogical association was noted during the study. Sepiolite tended to occur with dolomite and attapulgite with calcite. However, in a few cases, the sepiolite-containing samples had both carbonates; in a few other cases, only calcite occurred.

Analyses of samples from the lacustrine section investigated in detail (site 6, Fig. 1) revealed that attapulgite occurred only in the lower strata (e.g. Fig. 3) and sepiolite occurred mostly in the upper strata (e.g. Fig. 2). Small quantities of sepiolite were present in some of the lower strata. Discrete illite, discrete montmorillonite, and mixed-layered illite–montmorillonite occurred in variable amounts along with the sepiolite and attapulgite. Small amounts of kaolinite were often also present. Appreciable amounts of amorphous constituents were suggested in some samples because of poorly resolved X-ray diffractograms; however, no selective dissolution procedures were carried out. A soil caliche developed in "Cover sands" overlying the lacustrine section did not contain either sepiolite or attapulgite, but rather a clay mineral suite, typical of most soils of the area, i.e. discrete illite, mixed-layered montmorillonite–illite, and kaolinite.

Whenever a coarse-fine clay separation of sepiolite-rich samples was made, sepiolite invariably was concentrated in the fine fraction as exemplified in Fig. 2. This is in contrast to the reported concentration by Parry and Reeves (1968a) in coarse clay. A somewhat similar distribution between coarse and fine clay was noted when attapulgite-rich samples were separated; however, considerably more attapulgite remained in the coarse fraction than was the case in the sepiolitic samples. The relatively pure sepiolitic fine clay for which the X-ray diffractogram is shown in Fig. 2 was selected for additional studies, including CEC measurements, total chemical analysis, and electron microscopy. A CEC value of 42·2 meq/100 g was obtained. This value is higher than that ordinarily listed for sepiolite (Grim, 1968; Caillere and Henin, 1961), but corresponds to that listed by Rogers et al. (1956) for an aluminous sepiolite. Contamination with an expanding-lattice layer silicate could account for the higher value; however, in light of the K_2O content discussed below, expanding-lattice silicate content would appear to be minimal. Additional determinations on other samples will be needed before it is known whether this high value is typical of sepiolites in the area studied.

The chemical composition of the sample (Table 1) differs considerably from that of the Little Cottonwood, Utah sepiolite (Nagy and Bradley, 1955) in several respects: (1) The K_2O is much higher (exchangeable K should have been negligible since the sample was Na saturated), (2) Both SiO_2 and Al_2O_3 are appreciably higher, (3) MgO is markedly lower. If 6·5 is used as the average percentage K_2O in illite (Jackson and Mackenzie, 1964) then the sample would contain approx. 28% illite. This is rather surprising considering the electron micrograph (Fig. 4) and the X-ray diffractogram (Fig. 2); layer silicates do not appear to comprise such a large proportion of the sample. However, assuming there is 28% of layer silicates present, the high Al_2O_3 could be explained.

Table 1. Chemical composition of fine clay separated from sepiolite-rich sample

Constituent	% Composition
SiO_2	59·3
Al_2O_3	6·2
Fe_2O_3	3·0
CaO	0·8
MgO	11·9
Na_2O	0·1
K_2O	1·8
H_2O	15·0
Total	98·1

However, even when 28% of the SiO_2 is allocated to layer silicates, high SiO_2/MgO ratios still remain. The high ratios cannot be adequately explained with the present data; possibly considerable amorphous SiO_2 was present.

CONCLUSIONS

Our studies suggest that sepiolite and attapulgite were developed authigenically in alkaline lacustrine environments on the southern High Plains. Stratified high-carbonate units interbedded with those very low in, or devoid of, carbonates suggest arid periods of desiccation alternating with more humid periods. Such an environment would have obtained in a fluctuating Pleistocene climate. Millot (1970) has cited several examples of the "neoformation" of sepiolite and attapulgite along with carbonates and chert in alkaline lacustrine environments in different areas of the world. Wollast, et al. (1968) predict that sepiolite can form directly from dissolved silica and Mg^{2+} or that the waters can react with an earlier formed Mg-bearing phase. Since sepiolite and dolomite are more Mg-rich than attapulgite, Mg concentration would determine the mineral pair precipitated. The detailed study suggests increasing Mg concentration with time. The requisite silica could have been derived from widespread ash falls that, according to Evans and Meade (1944), occurred in the area during Pleistocene time. Ash lenses have been identified in most of the major lacustrine deposits that have been studied in detail, e.g. Blanco, Tule, as well as in a number of other localities in the area. These ash deposits should be more extensive in a playa lake than in the surrounding soils, since the water in a playa would tend to "capture" the ash. The Ogallala montmorillonite could also be the silica source as presumed by Parry and Reeves (1968a). Providing that our thesis is correct concerning the mode of origin of sepiolite and attapulgite in the areas studied, it seems likely that both minerals may be relatively common in surficial sediments and calcareous soils in warm and semi-arid regions. High concentrations of the minerals in such areas should suggest lacustrine deposits or contamination with reworked lacustrine material.

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Résumé — On a trouvé que la sépiolite et l'attapulgite sont des minéraux argileux communs, parfois dominants, dans les dépôts calcaires lacustres dans la partie sud des High Plains au West Texas et dans le partie est du New Mexico. Des débris de déflation ont dérivé des bassins et les sols calcaires développés dans les débris et dans les dépôts lacustres contiennent souvent aussi, soit l'un des deux, soit les deux minéraux. La dolomite est le carbonate communément associé avec la sépiolite, et la calcite a une relation analogue avec l'attapulgite dans les dépôts lacustres. La formation pédogenétique de la sépiolite et de l'attapulgite apparaît improbable dans la zone étudiée, puisqu'une association avec les matériaux lacustres a pu être établie dans un très grand nombre de cas.

On a trouvé la sépiolite hautement concentrée dans la fraction $< 0{,}2\,\mu$. Une distribution semblable, mais moins prononcée, a été notée pour l'attapulgite. Les études faites suggèrent que les minéraux se sont développés d'une manière authigène dans les environnements lacustres alcalins pendant les périodes de dessiccation. Un tel environnement, interrompu par des périodes plus humides, a pu être réalisé pendant des périodes sèches au Pléistocène. La cendre volcanique est proposée comme source possible de silice au départ. La concentration en Mg semblerait déterminer la formation soit du couple sépiolite–dolomite, soit du couple attapulgite–calcite.

Kurzreferat — Es konnte festgeltellt werden, dass Sepiolit und Attapulgit gewöhnliche, manchmal sogar die hauptsächlichen, Tonminerale in kalkigen Binnenseeablagerungen in den südlichen High Plains in West Texas und im östlichen Neumexiko darstellen. Winderosionsschutt aus den Becken und kalkige Böden, die sich in den Schutt und in den Binnenseeablagerungen entwickelt haben, enthalten ebenfalls häufig eines oder beide der Minerale. Dolomit ist das gewöhnlich dem Sepiolit zugeordnete Carbonat und Calcit hat eine ähnliche Beziehung zu Attapulgit in den Binnenseeablagerungen. Pädogenetische Bildung von Sepiolit und Attapulgit scheint unwahrseheinlich in dem untersuchten Gebiet, da eine Assoziierung mit Binnenseematerialen in einem sehr hohen Prozentsatz der Vorkommen erfolgte.

Sepiolit fand sich in hoher Konzentration in der $< 0{,}2\,\mu$ Fraktion. Eine ähnliche, wenn auch weniger ausgeprägte Verteilung wurde für Attapulgit festgestellt. Die Untersuchungen deuten darauf hin, dass die Minerale authigen in alkalischen Binnenseeumgebungen während Austrocknungszeiträumen entstanden sind. Eine derartige Umgebung, unterbrochen durch feuchtere Zeitspannen, hätte sich während trockener Pleistozänzwischenzeiten ergeben. Vulkanische Asche wird als Quelle des wesentlichen Quarz angesehen. Die Mg–Konzentration scheint zu bestimmen ob Sepiolit–Dolomit oder Attapulgit–Calcit gebildet wurde.

Резюме — Сепиолит и атапульгит являются общими, а иногда преобладающими глинистыми минералами в известковых лакустриновых отложениях южных высоких равнин в Западном Техасе и восточном Нью Мексико. Наносы пород от выветривания бассейнов и образующиеся в наносах и отложениях лакустрина известковые породы, очень часто содержат оба или один из этих минералов. Доломит, являющийся карбонатом, обычно связанный с сепиолитом и кальцитом, имеет подобное же отношение к атапульгиту в лакустриновых отложениях. Педогенное образование сепиолита и атапульгита не кажется вероятным в изучаемом районе, т.к. исследование связи с лакустриновыми материалами производилось в очень большом проценте случаев.

Сепиолит оказался очень концентрированным в $< 0{,}2\,\mu$ долях. Подобное, но менее выраженное распределеине было отмечено для атапульгита. Исследования предполагают, что минералы образовались аутигенно в средах щелочного лакустрина в периоды высушивания. Такая среда с периодами более влажных перерывов могла получаться во время сухих интервалов плейстоценовой эпохи. Предполагается, что вулканическая зола является источником основного кремнезема. Концентрация Mg как будто определяет образование либо сепиолита-доломита, либо атапульгита-кальцита.