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SMECTITE / ILLITE DISTRIBUTION AND DIAGENESIS IN THE SOUTH TIMBALIER AREA, NORTHERN GULF OF MEXICO

A Thesis

Submitted to the Graduate Faculty of the
University of New Orleans
in partial fulfillment of the
requirements for the degree of

Master of Science
in
The Department of Geology and Geophysics

by

Mark Dallas Dixon

B.S., Angelo State University, 1990

August, 2005

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Table of Contents

List of Tables

List of Figures

Abstract

Clays and clay mineral distribution studies are important for understanding the geological history of the Gulf of Mexico Basin, but few studies document any subsurface clay mineral distribution in the Gulf of Mexico.

Shale samples from nine wells (30 samples) in the South Timbalier protraction were selected near known paleontological markers identifying the Miocene, Pliocene, and Pleistocene boundaries. Bulk mineralogy of each sample, determined by XRD, is primarily mixed-layer smectite and illite with a minor amount of kaolinite. The mixed-layer mineralogies are end-member smectite, mixed-layer smectite, mixed-layer illite, and end-member illite.

These clay mineral fractions do not correlate with age. The illite mixed layer percentage correlates with depth, but the correlation decreases when depth is converted to temperature. However, the illite mixed layer fraction does not exhibit a strong correlation in this multi-well study when compared to a single well study in Ship Shoal using identical methods (Totten et al., 2002).

Introduction

Why would it be necessary to be concerned about clays in the Gulf of Mexico? What possible use would there be for an interest in clay mineral distribution? Is there a reason to map subsurface clays? Not only are these valid questions, but they are real issues for drilling in the Gulf of Mexico Basin. Not only is clay mineral distribution important for understanding the development the geologic and depositional history of the Gulf of Mexico (GOM), it is of particular interest for drilling operations. If an operator knows where to expect an expandable clay zone, the drilling process can be prepared to encounter it. It is important for operators and service companies to know what to expect or how to plan drilling a well in an area with swelling clays. The shales in the GOM are relatively young and can cause problematic drilling because of the amount of expandable clays that can be encountered. A swelling clay occurs when filtrates from drilling fluids, water based fluid in particular, enter the formation at the wellbore wall. The phenomenon of swelling is dependent on the chemistry of the fluid, the mineralogy of the shale, and the clay's chemical state within the shale at the time of contact. The most common swelling clays are smectite and mixed layer smectites. "Gumbo", a generic term for swelling clay formations, is commonly encountered in the GOM. Gumbo shales are soft and very sticky when wet, forming highly plastic masses which will adhere to metal surfaces. It will plug pipes, tools, flowlines, and shale shaker screens (Figure 1). Gumbo usually contains a large amount of smectite clays. A "gumbo attack" (Figure 2) can occur when drilling into a very reactive, smectite rich shale. It causes expensive rig time due to removal from the drill floor, flowlines, and

causes excessive circulation time to remove from the wellbore annulus. Other than being sticky and exhibiting severe swelling, it can also be dispersed into colloidal

Figure 1: Shale shaker removing drill solids from a borehole

Figure 2: An Example of a gumbo attack

sized solids and cause severe problems with typical solids control equipment, which has difficulty screening the colloidal sized solids out of the drilling fluid. Drill solids build up causes an increase in drilling fluid viscosity, which can cause excessive circulating density which, often times, will lead to lost circulation (less fluid volume coming out of the hole than what is going in). Another issue related to drilling clays and shales is sloughing. Wellbore instability due to the presence of sloughing and heaving shales is a function of overburden pressure, pore pressure, degree of water adsorption by clays, and local tectonic forces (Tschirley, 1978). Some factors that tend to promote sloughing include high content of high yield clays in shales, age of the shales, and high water wettability of penetrated rocks (Chilingarian, 1981). Severe hole enlargements and washouts are usually associated with sloughing and heaving shales (Figure 3), and is one of the factors that tend to promote hole instability. An interesting question related to zonal isolation that had an effect on this study asked, "Is the high pH of cement filtrate causing some sort of alteration of the clays in the shale, which in turn, reduces formation integrity, causing casing shoe washout or breakdown while cementing, or is it simply that the shales have bad formation integrity" (Harris, 2001).

The industry attempts to prevent drilling problems associated with shale. Drilling fluid laboratories prepare tests that simulate a drilling fluid's effect on a shale of interest. A simple, quantitative test was developed in the industry long ago to determine the initial effect of a fluid on a shale of interest. In brief, the labs test shales from offset wells near the wellbore of interest to design a fluid to inhibit its inherent clay swelling. Once the shale has been collected, it is pulverized and formed into a shale wafer on a

hydraulic press. The wafer is added to a fluid and rolled for a set time at a set temperature. The results are examined and if the shale maintains its integrity, the fluid and shale undergo more rigorous testing. An example of an inhibited shale is seen in Figure 4.

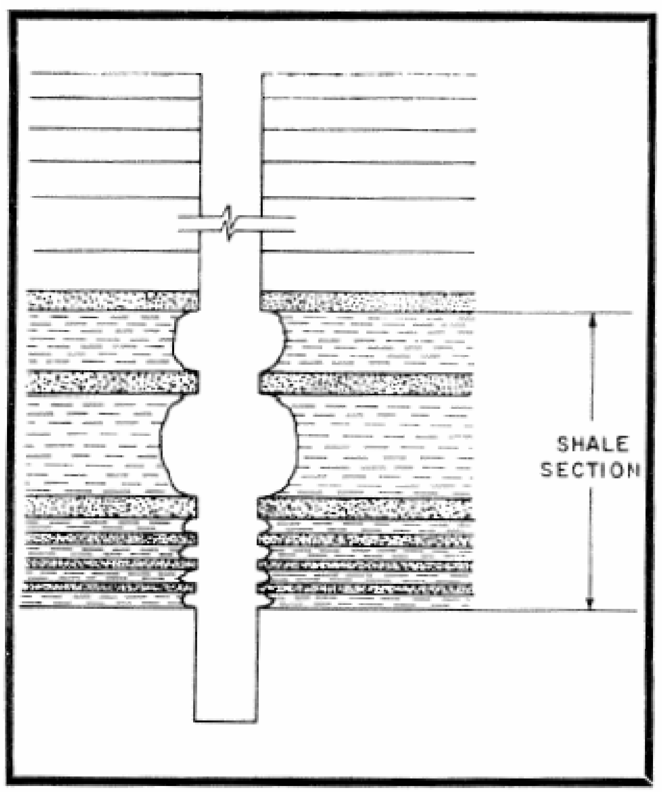

Figure 3: Wellbore washout due to enlargement caused by wellbore instability. Washouts are the enlarged, ovate sections of the borehole (Chilingarian & Vorabutr, 1981)

Figures 5, 6, and 7 are examples of shales that were not inhibited by a drilling fluid. An accretion test, a relatively new test that is currently being used in the industry, tests a shale's ability to stick or "accrete" to metal objects. It is primarily used to test bit balling (a drill bit with a mass of sticky, finely ground material attached to it), but can be used as a secondary test to determine inhibition. The test, in short, consists of a 3" X 1.25" metal pipe and shale. The pipe is weighed, and then rolled in a jar with 20 grams of the shale cuttings for 30 minutes at a set temperature. The tube is removed from the jar and dried in an oven for 30 minutes, and then it is reweighed. The difference in weight will determine the amount of shale accretion. The less accretion observed on the metal cylinder used in the test, the better the design of the drilling fluid used to inhibit shale hydration. Figures 8, 9, 10, and 11 are examples of an accretion test.

Abundant literature exists on clay minerals in the Gulf of Mexico, which include the smectite to illite concept proposed by Burst (1969) and the paper by Hower et al. (1976) which refined the idea of smectite to illite transformation with burial. Although there is abundant literature with respect to clay-mineral diagenesis, there have been few studies investigating regional clay mineral distribution in a geographic area in the subsurface Gulf of Mexico, except for the limited depths associated with piston coring, which only penetrate a few feet of surface sediment. The only study that investigated clay mineral distribution in the Gulf of Mexico focused on recent surface seafloor sediments (Devine, 1971). Studies of clay mineral distribution with respect to time examined samples from a single well (e.g. Aronson and Hower, 1976; Boles and Franks, 1979). The focus of this study is to investigate clay mineral distribution from multiple wells and multiple intervals of the wells from South Timbalier, approximately

1800 square miles in area, located in the Gulf of Mexico basin and to determine the location of swelling clays in the area that has a history of drilling related problems.

The variation of smectite-rich and illite-rich clays between different locations, depths, stratigraphic age, and temperature is examined in this research. The method developed to physically separate these mineral species by density used in a single well study in Ship Shoal (Totten, et al, 2002), was used in this study. This study will compare

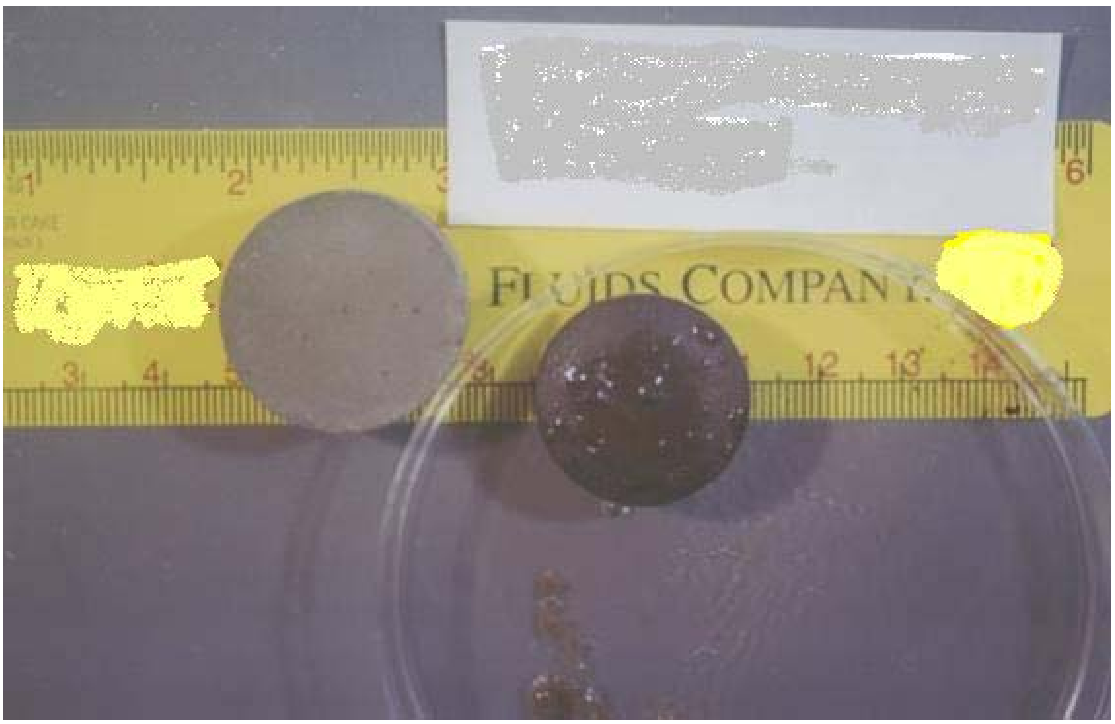

Figure 4: An example of an inhibited shale. Wafer on the right is the control reference. Wafer on the right is the sample that was exposed to a drilling fluid at a preset temperature and time.

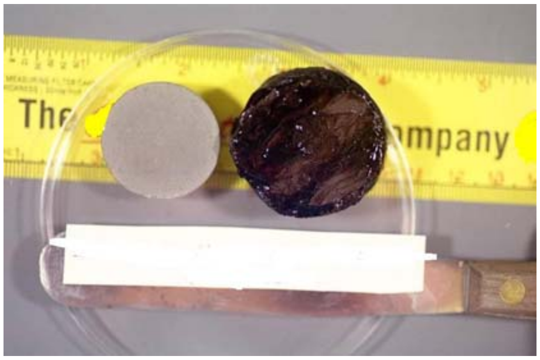

Figure 5: An example of an uninhibited shale, top view. Note the amount of swelling around the diameter of the shale wafer when compared to the control sample on the left

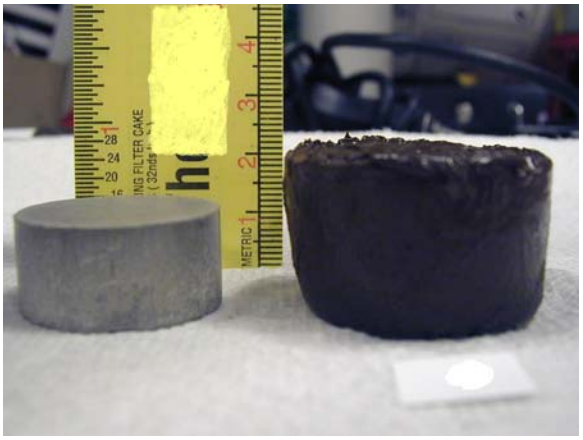

Figure 6: A profile view of an uninhibited shale

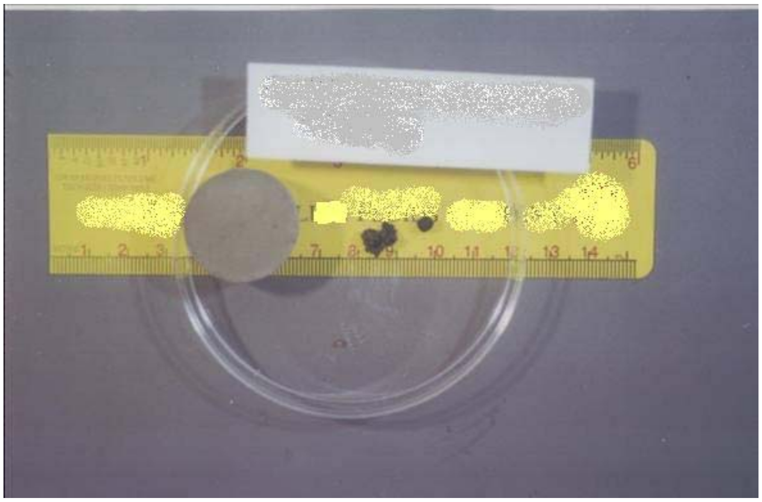

Figure 7: An example of an uninhibited, dispersed shale. Control Sample is on the left. Notice the absence of most of the shale that was exposed to a drilling fluid of interest.

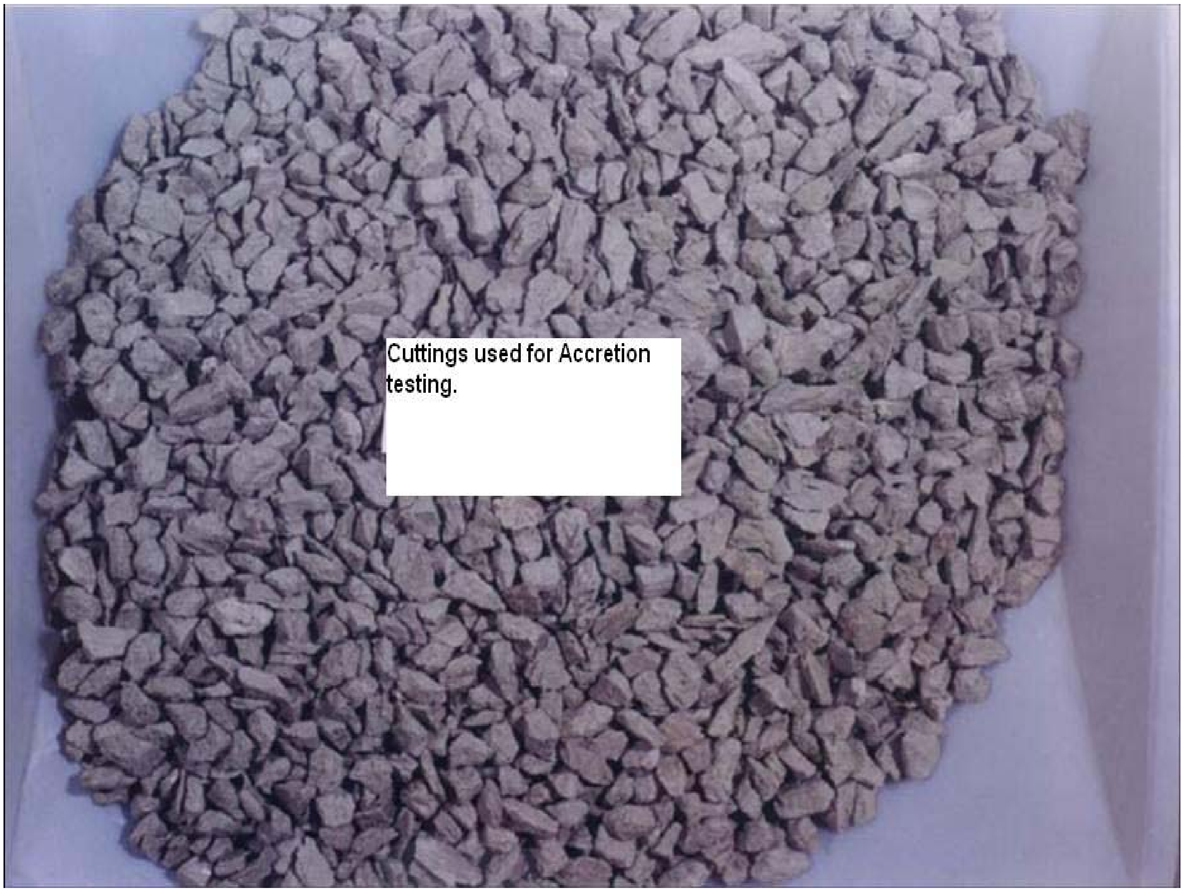

Figure 8: Cuttings used in an Accretion test. Cuttings are passed through a 5 mesh sieve to a 10 mesh sieve. Cuttings retained on the 10 mesh sieve are tested.

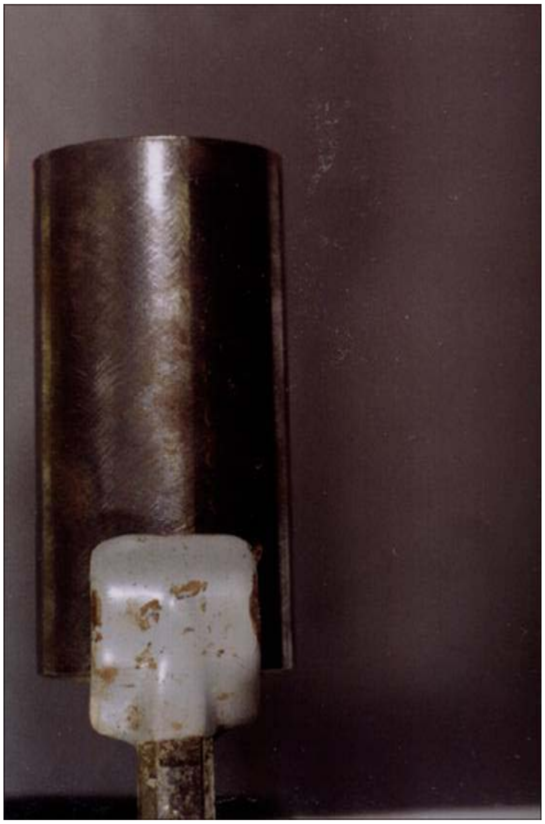

Figure 9: An example of a clean accretion tube

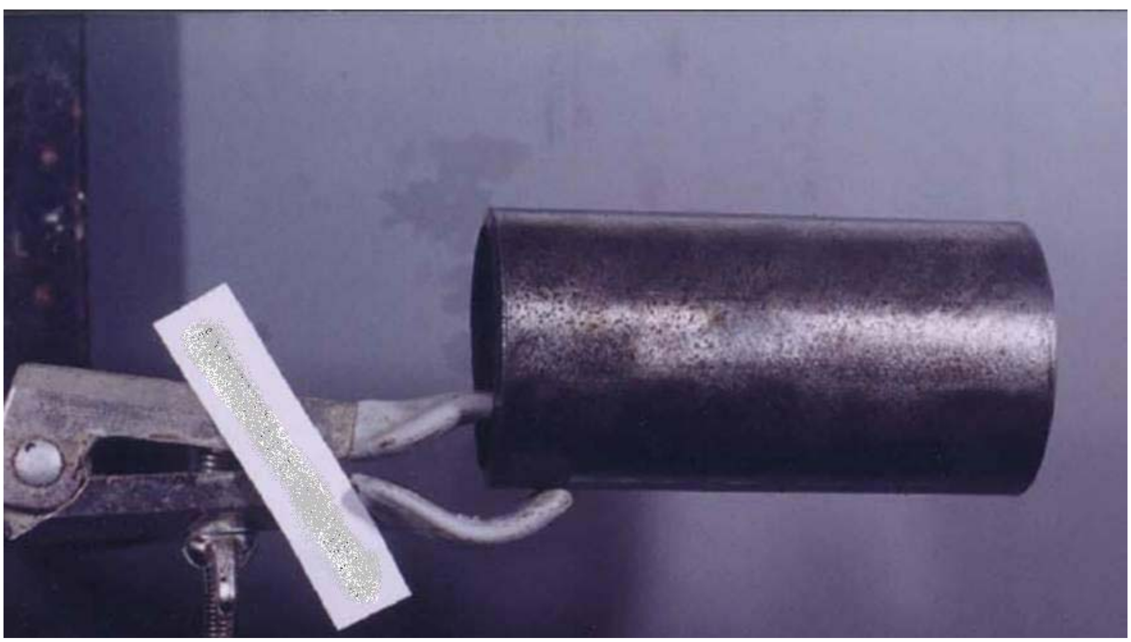

Figure 10: An accretion tube that was rolled with reactive shale / clay in an inhibitive mud. Note that the tube is just as clean as the tube in Figure 9. The fluid used in this test is very inhibitive and does not promote shale / clay hydration.

Figure 11: An example of a bad accretion test. Note the amount of shale cuttings that accreted to the pipe. The fluid used in this test does not inhibit shale / clay hydration.

and contrast the results of the Ship Shoal study with a wider distribution within the South Timbalier area.

Previous Research

Johns et al., (1954) investigated clay mineralogy of a river-bay-gulf system along the south Texas coast. They discovered that the Guadalupe River was introducing a dominantly montmorillonitic clay into bays, and within the bays, the montmorillonite content was said to decrease rapidly with an increase in chlorite and illite. These authors favored a diagenetic explanation for the change. Griffin and Johns (1958) investigated clay mineral relationships of surface and shallow cores from the southeast part of the Mississippi Delta. When the authors grouped clay samples according to the depositional environment, they noted that montmorillonite was slightly more abundant in three of four Mississippi River samples than in ninety-six samples from adjacent, more saline environments of the same age.

Weintritt and Fan (1957) investigated clay mineralogy of East Bay, Texas. They noted an abundance of montmorillonite inshore than in the more open part of the bay. They calculated clay mineral determinations on total weight of the sediment sample. The results indicated that as clay content increases, so does montmorillonite and all the other clay minerals. The same general clay mineral suite is present throughout the entire bay.

Milne and Early (1958) studies clay mineral distribution in the lower Mississippi River, Mississippi Delta, Mississippi Sound, Mobile Bay, and the inshore areas of the Gulf of Mexico. They found that montmorillonite is the predominant mineral being supplied by the Mississippi River and that the clay mineral suite shows no significant

change as it enters the saline environment. Along the shelf edge, they discovered more illite, which was attributed to clay alteration in an area of reduced sedimentation. The authors found more kaolinite to the east, in particular the Mississippi Sound and Mobile Bay, than in the Mississippi Delta.

McAllister, Bader, and Kunze (1958) studied clay mineralogy of thirty-six grab samples and three cores from the western side of the Mississippi Delta. They found that the clays of that area consist of about 55% montmorillonite, 25% illite, 25% kaolinite, and no chlorite, even though a thorough search for chlorite was made. They found little diagenetic change and determined diagenesis occurred too slowly to be of much significance.

Devine (1971) determined that diagenesis by clay mineral formation or transformation does not have a major effect on the mineral distribution patterns of the Gulf of Mexico. The author investigated the upper 10-cm or less of several surface samples of the Gulf of Mexico floor. He determined that six major minerals could be recognized by x-ray diffraction. The average bulk density is 28% illite, 23% calcite, 19% quartz, 15% smectite, 10% chlorite, and 7% kaolinite. Quartz and calcite make up a small percent of the <2-micron fraction.

Meyer (1958) examined clay mineral distribution in two long cores and 33 surface samples from all parts of the Gulf of Mexico. The clay mineralogy of the two cores was dominated by montmorillonite and with small amounts of illite and chlorite. The second core came from the Sigsbee Deep and showed, more or less, the same mineralogy as the slope core, except that mixed layering was more common and illite was more

abundant. Mixed layer mica/montmorillonite persisted to a depth of 1 meter, where it disappeared.

Slatt, et al., (1992) examined well cuttings from a cross section that extended from the Ouachita tectonic belt in southern Arkansas to High Island, northern Gulf of Mexico. The clay mineral assemblages encountered included mica, kaolinite, smectite, chlorite, and mixed layer illite/smectite. Illitization was observed as cuttings depth increased. Smectite did not occur below 3,230 meters.

Generally all previous studies, except Slatt et al., examined the top 1-meter or less of sediment. Unfortunately, both surface and sub-surface clay mineral distribution studies just don't have enough data and often have contradictory results. Studies of clay mineral distribution patterns of recent sediments are incomplete. Often, research does not take an entire sedimentary basin into account to allow all factors to be properly evaluated (Griffin, 1962).   The conclusions that were drawn from previous clay mineral distribution studies were not the only conclusions possible. A common problem with plotting clay distribution patterns is accounting for foreign clays in a sedimentary basin. Research results often conclude that the only system to supply the offshore depositional area is a single river. Other sources, in particular, other river systems may have made a significant depositional donation, but were not considered.

Rivers supply most of the clay minerals in the northern Gulf of Mexico (GOM). The Mississippi River contributes most of the montmorillonite in the northern GOM while smaller rivers contribute limited amounts of illite, chlorite, kaolinite, and montmorillonite. The GOM average bulk density is 28% illite, 23% calcite, 19% quartz, 15% smectite, 10% chlorite, and 7% kaolinite , where quartz and calcite make up a small percent of

the <2-micron fraction.   Clay mineral distribution is dependent on delta migration (Whynot, 1986).

Geologic Setting

The Gulf of Mexico is the largest semi-enclosed depositional basin in North America (Coleman et al., 1991), and is classified as the ninth largest ocean in the world, a “remnant of one or more extensive seas” (Phleger, 1951). The northern GOM is bordered by continental shelf and slopes toward the basin. The westside of the GOM is a narrow and steep coastal plain. It rises along the Campeche and Florida submarine escarpment to the Yucatan and Florida Carbonate platform in the south and east, and the Sigsbee escarpment bounds to the north and the deep central plain of the GOM.

The GOM lies in a semi-enclosed basin. The Gulf Coastal plain topography and geologic features influence the courses of the rivers that discharge into the GOM.   The innermost edge of the coastal plain is primarily Tertiary and upper Cretaceous sedimentary strata.   This segment provides the greatest variety and volume of sedimentary particles to the Gulf Basin (Poag 1981). The physiography of the GOM was also influenced by glacial and interglacial episodes during the Quaternary. Although ice sheets did not reach the Gulf, the climate, sea level changes, and meltwater influx affected the drainage systems and sediment supply to the GOM. Periods of increased glaciation produced periods of low sea level. Some areas that were submerged became dry land and were exposed and affected by erosion and climate. The morphology of the modern continental shelf has been primarily controlled by three things: sea level fluctuations during the Pleistocene, sedimentary deposition from changing courses of the Mississippi river, and tectonics associated with diapirism.

River Drainage into the GOM

Three major rivers supply most of the detritus to the northeastern GOM (Griffin, 1962). The Chattahoochee-Appalachicola River system of north Florida and the Alabama-Tombigbee-Mobile River system of Alabama provide the primary drainage to the east. These rivers arise in the Appalachians upland of Alabama and Georgia. The Mississippi River flows through the middle of the coastal plain and enters the Gulf in a huge bird foot delta at the southern tip of Louisiana. The Mississippi River drains 41% of the United States from the heartland between the Appalachians and the Rocky Mountains. The Mississippi River contributes about 600 million tons of sediment and has supplied sediments to the Gulf of Mexico since the late Jurassic, "constantly feeding sediments to the receiving basin and building a thick Jurassic, Cretaceous, Tertiary, and Quaternary sequence of inter-fingering deltaic, near shore coastal brackish water, and marine sediments, which have prograded the coastal plane shoreline seaward" (Coleman, et al, 1991). Mesozoic and Cenozoic deposits are estimated to have a total thickness in excess of 15 km (Martin and Bouma, 1978).

The physiography of the gulf floor determines the distribution and final location of deposits and sediments by coastal rivers. Seaward of the bays, estuaries, lagoons, swamps, and river deltas, three broad, gently-sloping segments of the continental shelf extend out to about 200 m depth. These include: The west Florida shelf, Texas-Louisiana shelf extending from Mobile Bay to the Rio Grande, and the Campeche shelf. The continental slope of Texas, Louisiana and Campeche is rugged. Numerous submerged banks, ridges, domes, and basins have been formed by intruding subsurface masses of salt.

The distribution of clay and other sediments in the GOM is related to the coastal and submarine physiography, climate, currents, and composition of coastal rocks. The primary source of siliclastic sediment is the Mississippi River. Most of the detritus of the Mississippi River is rich in silt, clay, and organic detritus. It is deposited in the deepwater of the GOM, and supplies the Central Gulf with silt and clay. Some of the Mississippi sediment load moves westward along the Texas-Louisiana shelf and forms a muddy substrate. The rivers along the Texas coast provide muddy siliclastic sediments. The Rio Grande supplies a mineralogically distinctive siliclastic sediments that form narrow bands of muddy deposits basinward of its mouth. The remaining siliclastic sedimentary regime borders the eastern Gulf shore of Florida. A few rivers drain the Pleistocene and upper Tertiary coastal plain rocks as far south as Charlotte Harbor, but provide only small amounts of clay and silt, therefore, the substrate is chiefly sand. Organic detritus in sediments of the western gulf is sparse in near shore areas (~0.5% of sediment), reaches a maximum on the upper slope (+/- 2%), and then decreases to +/- 1 % on the Sigsbee Plain. Clays are generally richer in organic content than sands. On the shelf, the organic matter (50%) is derived from land plants, but the origin of organic matter in the Sigsbee Plain is primarily of marine origin. The Mississippi Fan contains higher percentages of terrigenous organic debris than other bathyal and abyssal regions of the Gulf. Other Pleistocene carbonate deposits can be on banks of Texas, Louisiana, Alabama, and Florida.

Offshore Environment

Environments of deposition (EODs) of the northern and western edge of the GOM can be considered terrigenous while the south and southeastern edge of the

GOM is considered carbonate deposition. These EODs have not changed since the late Jurassic – early Cretaceous. Most of the sediment was delivered to the northern Gulf during the Cenozoic. Offshore Louisiana, the shelf is variable in width (the area of study), and is less than 20 km wide of the active mouths of the Mississippi River delta and in excess of 180 km of the western Louisiana coast. The section of Louisiana shelf in front of the Mississippi river experiences high sedimentation rates, generally in excess of 1m/year right of the mouth. These high sedimentation rates result in unconsolidated sediments and instability of the shelf, regardless of its gentle slope of less than 0.5 degrees. West of the Mississippi River delta, the shelf is broad, covered in mud, and is relatively flat. Holocene muds in the western gulf vary in thickness, but can be up to 10m and these muds were delivered by a slow westward drift from the Mississippi River. Offshore central Louisiana, shoals are composed of reworked sands from transgressed delta facies.

Cyclical sedimentation rates are complex in this region of the GOM. Sedimentation during low sea level is characterized by: variable thickness, rapid accumulated sequences, coarse grained clastic deposits (including sands and gravel), well defined depositional trends, and a wide variety of seismic responses (Coleman et al., 1998). Eroded channel systems formed during the last low sea level were that were cut by the Mississippi river and coastal streams in response to lowered sea levels. Quaternary deposits in the Northern GOM show a complex distribution pattern that has been influenced by changing sea level and shifting sites of deposition by the Mississippi River and are characterized by sedimentation associated with the constant offshore and

onshore shifting of depositional sites and by transgressions and regressions associated with sea level changes.

Stratigraphy

The Cenozoic stratigraphic section of the GOM is composed of very thick, laterally variable, sequences of sandstone, siltstone, and shale that are difficult to break down into distinctive units such as groups, formations, etc., and are difficult to map over large areas. When oil was discovered in the GOM basin, drilling revealed that sediments thicken in the subsurface, and that non marine rocks become marine in the subsurface. Unfortunately, Cenozoic nomenclature for the GOM does not follow the standard rules of stratigraphic nomenclature and lack adequate definition for formations, members, and so forth. Stratigraphic zonation of the GOM is largely dependent on paleontology, especially benthonic and planktonic foraminifers, calcareous nannoplankton, and some ostracodes. In some areas, parts of the Cenozoic are referred to as the Miocene, Pliocene, and Pleistocene. Many correlations are unreliable in the GOM because of changes in depocenter, structural changes, shifting deltaic sources, and changing rates of deposition. Modern depositional patterns of the Gulf of Mexico include terrigenous deposits that dominate the northern and western section of the Gulf, and carbonates across the platforms of the east and Southern Gulf since the late Jurassic. In the late Jurassic, sediment load overwhelmed the mostly carbonate environments because of the tectonically elevated western and northern interior of North America. The Central portion of the GOM basin is dominated by the Mississippi embayment. This influences the coastal plain from Alabama to east Texas, and consists of Mesozoic and Cenozoic rocks. There has been a gulfward shift of

depocenters from older to younger Cenozoic units, e.g., during periods of high sediment supply, the main sand depocenter shifted to the continental shelf edge where large quantities of sediments were delivered to the slope and basin floor. During periods of shelf flooding, the main sand depocenter shifted landward with little coarse sediment reaching the basin or reduced sediment supply.

Quaternary deposits of the GOM are as thick as 3600 meters and accumulated under the shelf between Texas and Louisiana. Around 3000 meters of Quaternary sediment has accumulated in the Mississippi Fan. Sediment beds of considerable thickness have accumulated in the northern part of the GOM basin within intraslope basins. Sedimentation and morphology were affected by frequent changes in climate, vegetation, drainage patterns, discharge characteristics, and sea level change during the Quaternary. The major effect of climatic fluctuations was the change in sea level. The lower sea levels exposed areas of former continental shelf which also lowered the base level of streams and rivers draining into the GOM. Lower sea levels caused seaward building of river deltas, which built out the continental margin. When sea level rose, coastal plains were submerged. The frequencies, rates, and magnitude of sea level changes during the Quaternary are not well established, but isotopic studies in the GOM basin have shown high frequency fluctuations within the sediment column. These changes in sea level, glacial meltwater discharge, and changing sites of deposition have influenced the sedimentation style in the Northern GOM.

The GOM basin extends from the coastal plains sediments onshore USA southward to the Sigsbee Escarpment and extends east west from the Florida Peninsula to the East Mexico Shelf. The Gulf Coast basin is rimmed by areas of the

Ouachita and Appalachian orogenic belts, and the basin lies a few miles offshore from the present shoreline.

Rocks deposited in the Gulf coast basin are primarily Mesozoic and Cenozoic in age. These rocks become younger southward. Sedimentation began in the Triassic with the deposition of red beds. This was followed by deposition of Triassic and Jurassic salt. Lower cretaceous carbonated developed in the western area of the basin and siliclastic sediments were deposited in the east. Upper Cretaceous sediment includes shales and chalks. Tertiary rocks are mostly siliclastic and were deposited in deltaic and deepwater environments. Miocene deposits are thick under the Louisiana coast with Pliocene depocenters under the central shelf and Pleistocene depocenters under the outer shelf and Upper slope).

South Timbalier is located in the Gulf of Mexico basin, southwest of Grand Isle LA, and is part of the continental shelf. The shelf gently slopes seaward and lies in fairly shallow water. The shelf has a slope of less than 1 degree over most of its extent. The seafloor surface around these blocks is generally smooth. South Timbalier is located in the northern Gulf of Mexico, approximately 15 miles southwest of Grand Isle. The most common structural process that occurs in this region is salt tectonism, and is closely related to deposition. Salt withdrawals form basins for sediment deposition. Thick sediment piles also occur on the sides of salt diapirs, in particular, on the downthrown sides of the diapir associated faults. Many of the structures in the area are salt diapirs. The depositional environment consists of sheet and amalgamated sheet sands, channel fill sands, or thin sands encased in overbank shales (Weimer et al., 1998). The continental slope and outer shelf are characterized by channel knolls,

ridges, and basins (Stude, 1978). The Cenozoic section of the Gulf Coast geosyncline represents a regressive wedge of offlap deposits "interfingered" with transgressive deposits. A westward shift of the Mississippi depocenter combined with a drop in sea level limited the South Timbalier sediment supply, encouraging shelf margin retrogradation, entrenchment and gorge development. Structural highs and lows existed throughout the Pliocene and early Pleistocene and was contemporaneous with sedimentation (Mason, 1992).

A South Timbalier cuttings database was utilized for this research. Chevron's data base covering South Timbalier has in excess of 4,200 samples to collect and examine, which are held at the UNO-Chevron Earth Science Laboratory at Harbor Circle, located in New Orleans, Louisiana.

Methods

Sample wells were chosen based upon their geographic distribution in the South Timbalier Protraction area (Fig. 12) and the availability of well-cuttings at the UNO-Chevron Earth Science Laboratory. Sample Depths were chosen using paleontological markers that correlate to the Miocene, Pliocene, and Pleistocene boundaries, reported from the public paleontological information database of the Minerals Management Service (available online at: ww.gomr.mms.gov/homepg/pubinfo/pdfindex.html). Shale-rich intervals were chosen for sampling as indicated by well logs. Rock chips were examined and handpicked from 9 wells that contained 30 sampling depths within South Timbalier. Sand-rich and coquina rich sections were not used in this study. The Shale chips were crushed using a mortar and pestle and passed through a 62 μm sieve, (0.0025 inch, n0. 230 screen, Tyler equivalent 250 mesh). The methods developed by

Hanan and Totten (1996), were used to separate the clay species. This method is efficient in separating smectite from illite.

A small sample of powdered shale was mixed in 15 ml distilled water and was agitated with an ultrasonic probe for approximately 5 minutes. After ultrasonic agitation, the sample of disaggregated cuttings suspended in distilled water was filtered through a 0.45 Millipore filter paper. The filter paper and sample filter-cake was mounted on a 25 mm glass disk using standard's glue diluted with distilled water. This process was performed on all thirty samples. Samples were allowed to dry 24 hours. Excess filter paper was carefully trimmed from the glass disks. Samples were placed in a desiccation chamber using Dry Rite as the desiccation medium. Samples were x-rayed and data was downloaded. Samples were then glycolated for 12 – 24 hours and x-rayed again with same angles of deflection and data was downloaded.

LMT Preparation

Lithium metatungstate is an inorganic water based non-toxic salt. The specific gravity of LMT can be adjusted using distilled water to between 1.0 and 3.4 at room temperature, 25 deg C.

Rheological characteristics of LMT:

a. 4cP @ 2.3 g/cc
b. 20 cp @ 2.7 g/cc

LMT can be recovered by evaporation at low temperatures, less than 100 deg C and reused. According to Totten, et al. (2002), it is important not to let the LMT recrystallize into a solid form because it is difficult to dissolve. To avoid this problem, the LMT was

evaporated in a dynamic state by evaporating water on a hotplate stirrer with a magnetic stir bar until the LMP appeared dark, viscous, and water-free.

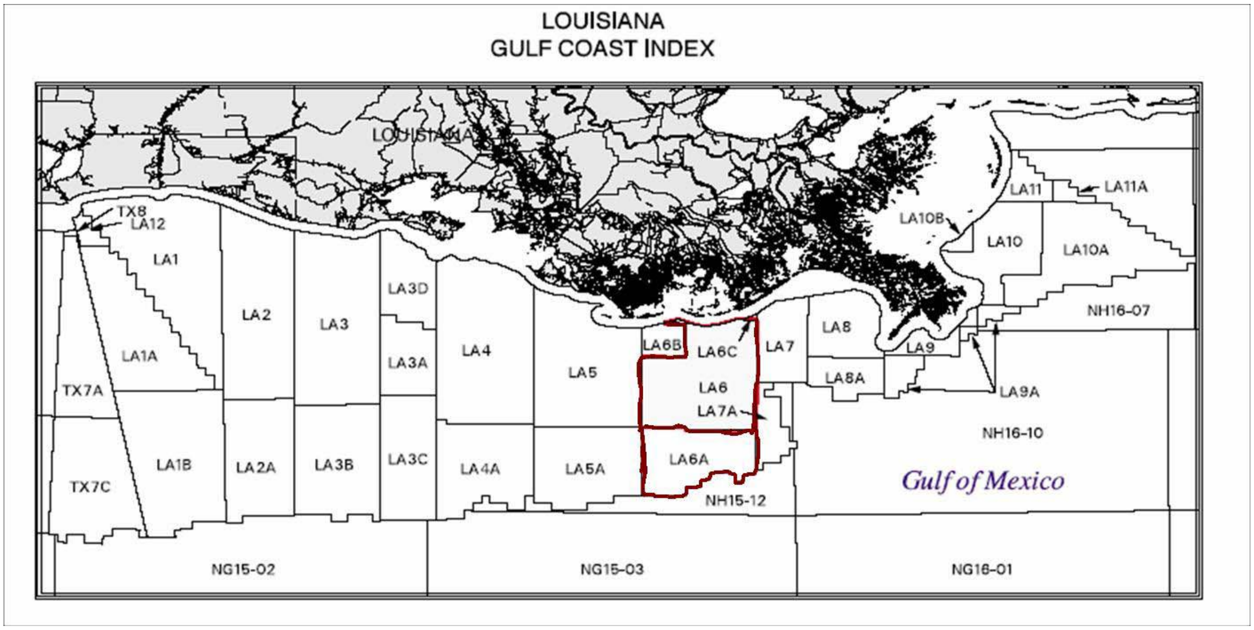

Figure 12: Protraction of offshore Louisiana. South Timbalier outlined in red.

To obtain an LMT solution of a specific gravity, a stock solution of 3.1 g/cc is diluted by adding distilled water. LMT density was determined by using the following formula: Vw=(VoPf – VoPo)/(PwPf), where

A: Vw=volume DI water

B: Vo= volume of stock solution

C: Pf = final density required

D: Po = density of stock solution (3.1 g/cc)

E: Pw= density of water at lab temperature.

Samples were weighed out after cutting and pulverizing the shale samples into powder. Approximately 1 gram of ground, disaggregated shale cuttings was placed into 50 ml polycarbonate centrifuge tubes with screw top lids. Distilled water was added to balance the weight of the tubes (this prevented the centrifuge from becoming out of balance while operating at high speeds). The mixture was suspended by shaking the tube vigorously. All of the suspension easily dispersed in the distilled water. The tubes were centrifuged at 3000 rpm for one hour at a constant temperature. All particles sank in the DI water separation. The Distilled water separation was used as the base for XRD bulk mineralogy and clay mineral fraction study.

LMT density was checked with a 25 ml pycnometer on a Denver Instrument digital scale. Precise density determination was determined by weighing the LMT required to fill a 25 ml pycnometer. Working LMT solutions were stored in air tight flasks. 2.3 g/cc LMT was added to fill and balance the weight of the tubes. To minimize rafting, defined by Totten et al, (2002) as, "the dense sink material trapped in a lighter fraction", the float at the top of the tube was resuspended without disturbing the sink

portion by gently swirling by hand until a “suspended solids cloud” was formed. The tubes were then recentrifuged at 3000 rpm for an additional hour. The centrifuged tube containing the desired LMT density was frozen in a six fluid ounce liquid nitrogen bath. The thin layer of frozen float material at the top of the tube was removed by washing with DI water into a 0.45 acetate filter apparatus (Pell type negative pressure filtration apparatus) and recovered. The float layer includes most of the material with the desired density or less of all size fractions. As per Totten et al (2002), “some of the finest sized material … has not had time to float to the top of the tube, but all of the material greater than desired density has had time to sink through the small distance at the top of the tube.” The liquid above the layer of sink material was slowly withdrawn with a transfer pipette and set aside. The purpose of this step was to analyze the ultra-fine colloids in the suspended portion of the prepared LMT solutions. The LMT solution with entrained solids is referred to as “suspended solids”. The remaining LMT was allowed to “melt” at room temperature over time.

Typically, the liquid LMT between the sink and float layers appeared to be clear and solids free however, according to Totten et al (2002), the calculations based upon Stoke’s Law suggest that this liquid will still contain ultra-fine material, especially of particles with a density near that of the LMT. The layer in the bottom of the centrifuge tube only contained material that had a density greater than 2.3 g/cc, of all size fractions. For the 2.4 g/cc, separations, the 2.4 g/cc LMT solution was added to the tube that contained the 2.3 g/cc sink material. The sink material was resuspended in the 2.4 g/cc fluid using an ultrasonic probe for approximately five minutes, which completely disaggregated the material. The tube was centrifuged as before to isolate

the material that had a density greater than 2.4 g/cc. This process was repeated using 2.7 and 2.85 g/cc LMT. The final sink greater than 2.85 g/cc was recovered by filtering across a 0.45 micron filter paper. This fraction contains heavy minerals. The result of the density separations is four clay mineral bearing density fractions less than 2.3 g/cc, 2.3 g/cc to 2.4 g/cc, 2.4 g/cc to 2.7 g/cc, and 2.7 g/cc to 2.85 g/cc. Densities greater than 2.85 g/cc are separated waiting on further evaluation. The amount of sink at each density was determined by centrifuging for one hour at 3000 rpm. Suspended LMT was filtered to determine amount of float material present in each sample separation.

XRD analysis

The mineralogy of the separated clay fractions was determined by X-ray diffraction (XRD) at the Microbeam Laboratory in the department of Geology and Geophysics at the University of New Orleans. Oriented samples mounts were prepared after the method of Moore and Reynolds (1997) for each LMT separation. (The 2.3 g/cc, 2.4 g/cc, 2.7 g/cc and 2.85 g/cc LMT float material separations). Bulk mineralogy analysis (no LMT separation) compared air dried to glycolated samples, but no other air drying was performed after that because of extended tube life usage of the X-ray diffractometer (I anticipated burning out the tube with twice the amount of x-ray analysis). Glycolated analysis of the sample preparation were made on a Scintag XDS-2000 diffractometer using $CuKa1_1$ radiation, at 40 kV and 20 mA, scan range 2-40 deg, step size 0.02 deg, and dwell time of two seconds per step. Glycolation was achieved by placing samples in a glycol-saturated atmosphere for 24 hours at ambient temperature. Clay standards from the Clay Mineral Society were not compared in this round of tests.

Definition of Mineral Separates:

Mineral separates are defined in the following manner:

EMS: end member smectite, <2.3 g/cm3 density fraction

SML: Density fraction between 2.3 and 2.4 g/cm3, termed smectite rich mixed layer

IML: Density fraction between 2.4 and 2.7 g/cm3, termed illite rich mixed layer

EMI: fraction with density > 2.7g/cc, named end member illite.

Results

It is not physically possible to separate the shale into a single mineral component, but, the LMT method is efficient enough to concentrate the major component of the shales collected in South Timbalier. Figure 13 illustrates the four XRD clay mineral components separated from a single sample, comparing an XRD pattern of the un-separated clay-mineral fraction. Each separate has its own unique mineralogy, composed, primarily, of the mineral phases named below. The density separates are not mono-mineralic, and the names of each separate are not rigidly applied as an accurate description of the mineralogy in every case in this study, and differ in many respects from the same density separates of the earlier study. The names of each separate are used for easy comparison between the two studies.

The four mineral separates, defined in by Totten et al. (2002) and the different amounts of each component for each sample location and depth are given in Table 1. The geologic age and corrected temperature data derived from bottom-hole temperature are data reported on the well logs.

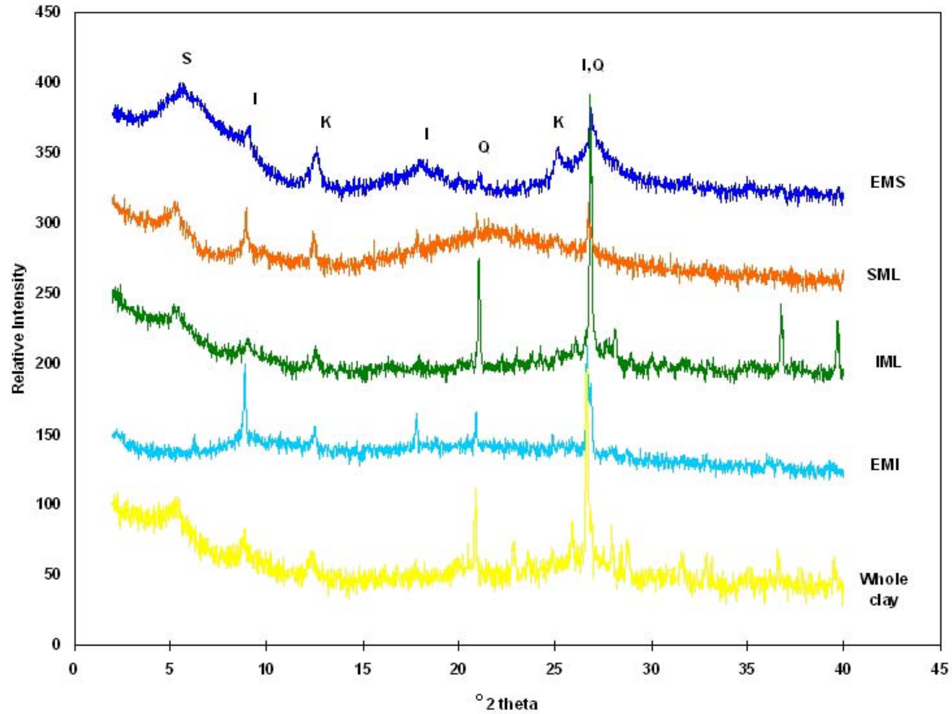

Figure 13: Four different mineral components separated from a single sample. The Whole clay sample is included for comparison.

Table 1. Sample location, depth, age, and density separation data.

Sample	% EMS	% SML	% IML	% EMI	Depth	Age	OCS-G#	STBlock
1	32.12	39.66	26.45	1.78	5470	Plio	0615#F3	22
2	26.15	36.86	33.90	3.09	5700	Plio	0615#F3	22
3	2.52	45.13	50.40	1.94	10380	Mio	0615#F3	22
4	6.52	55.53	36.91	1.04	4570	Pleist	2927 #1	59
5	4.62	47.95	46.68	0.75	8931	Plio	2927 #1	59
6	10.48	41.94	46.33	1.25	9080	Plio	2927 #1	59
7	0.55	26.08	71.41	1.96	14210	Mio	2927 #1	59
8	7.91	41.14	48.06	2.89	10180	Pleist	1559 #2	111
9	2.38	36.82	58.93	1.88	11100	Plio	1559 #2	111
10	0.71	34.39	64.05	0.85	11230	Plio	1559 #2	111
11	0.53	23.99	73.28	2.20	14170	Plio	1559 #2	111
12	2.18	27.68	68.79	1.34	14030	Plio	1247#10	160
13	0.93	31.15	65.68	2.23	14150	Plio	1247#10	160
14	14.79	51.63	31.14	2.44	12140	Plio	1248#C4	161
15	20.23	40.27	38.03	1.47	12260	Plio	1248#C4	161
16	2.63	49.37	46.74	1.25	14814	Mio	1248#C4	161
17	0.92	26.13	71.47	1.49	12650	Plio	1960 #1	148
18	0.98	25.15	71.88	1.99	12740	Plio	1960 #1	148
19	56.76	3.52	36.19	3.52	9410	Plio	1265 #3	196
20	9.05	24.60	64.14	2.20	9500	Plio	1265 #3	196
21	11.08	31.93	55.66	1.33	11480	Plio	1265 #3	196
22	19.88	23.20	54.27	2.65	12470	Plio	1265 #3	196
23	22.45	46.17	30.05	1.33	7610	Pleist	1575 #2	205
24	15.22	31.95	52.02	0.82	7700	Pleist	1575 #2	205
25	4.29	45.56	49.10	1.04	11720	Plio	1575 #2	205
26	5.61	51.01	41.36	2.03	11840	Plio	1575 #2	205
27	0.29	55.71	42.50	1.50	14000	Plio	1575 #2	205
28	0.78	49.27	46.87	3.07	7780	Pleist	2154 #2	314
29	3.17	63.26	30.28	3.29	8260	Plio	2154 #2	314
30	0.43	50.32	44.19	5.05	8380	Plio	2154 #2	314

Discussion

Thirty samples were collected from a wide geographic distribution in South Timbalier, extending from Block 22, which is near the shoreline to Block 314, near the outer continental shelf. Each sample collected was weighed, glycolated, and x-rayed. After the density separations and XRD analysis, the samples were compared to a variety of factors that may have affected the individual sample mineralogy. These factors include: bulk comparison of clay mineralogy to depth, age, and temperature, as well as the individual sample blocks compared to depth, age and temperature. Sample depth and age are sometimes synonymous with one another, especially in these discussions. For example, <u>South Timbalier Block 22 v. Depth</u> would yield the same results as <u>South Timbalier Block 22 v. Age</u>. With such a small sample set of ages in a wellbore, there are instances where only one age may be present. A good example of this would be South Timbalier Block 22, which has two Pliocene samples at 5470' and 5700' respectively and one Miocene sample at 10380'. No valid statistical data can be obtained with only one Miocene sample in one wellbore, and a straight trend line would be observed with only two Pliocene samples in the same wellbore. Therefore, it would be better to treat the analysis of this example with respect to depth rather than attempting to compare mineralogy with respect to age *and* depth.

Illitization

The results of this multi-well study in South Timbalier are similar in a number of respects to the results that were reported from the single well study in Ship Shoal (Totten et al., 2002). Illitization occurs at the expense of smectite-rich layers as expected from results published in previous studies. Figure 14 illustrates this for the

illite-rich mixed-layer component (IML) of this study. IML in this study correlates with depth (correlation coefficient r= 0.62) and is similar to the results (Figure 15) from the single well study in Ship Shoal (r=-0.89). Both studies support the well-publicized control of depth on illitization. Many diagenetic variables are dependent on depth; e.g., geothermal gradient, burial history, rate of deposition, proximity to salt bodies, and fluid migration. These variables are minimized in a single study in a single wellbore, which is, in comparison to this study, one small area. The correlation coefficient in this multi-well study compared to the single well study with respect to depth indicates a lower correlation coefficient. In other words, a single wellbore will have a more consistent diagenetic history than a series or group of wellbores in a wide geographic area. The correlation between illitization and bottom-hole temperature was examined, which is generally considered the primary control on illitization (Boles and Franks, 1979). However, this is not seen in this multi-well study, as the correlation between the percentage of the illite-rich mixed-layer component (IML) and calculated bottom-hole temperature is less (r=0.47). The overall diagenetic history each sample experienced during burial is better reflected with depth than with current bottom hole temperature alone. The calculated bottom hole temperatures from current / recent well logs may not reflect the temperature histories that were driving clay-mineral transformations.

Although the percentage of IML present increases during diagenesis, this fraction exhibits consistent mineralogy in each sample as seen in Figure 16. The mineralogy of the IML fraction is consistently an illite-dominated mixed-layer clay regardless of depth encountered.

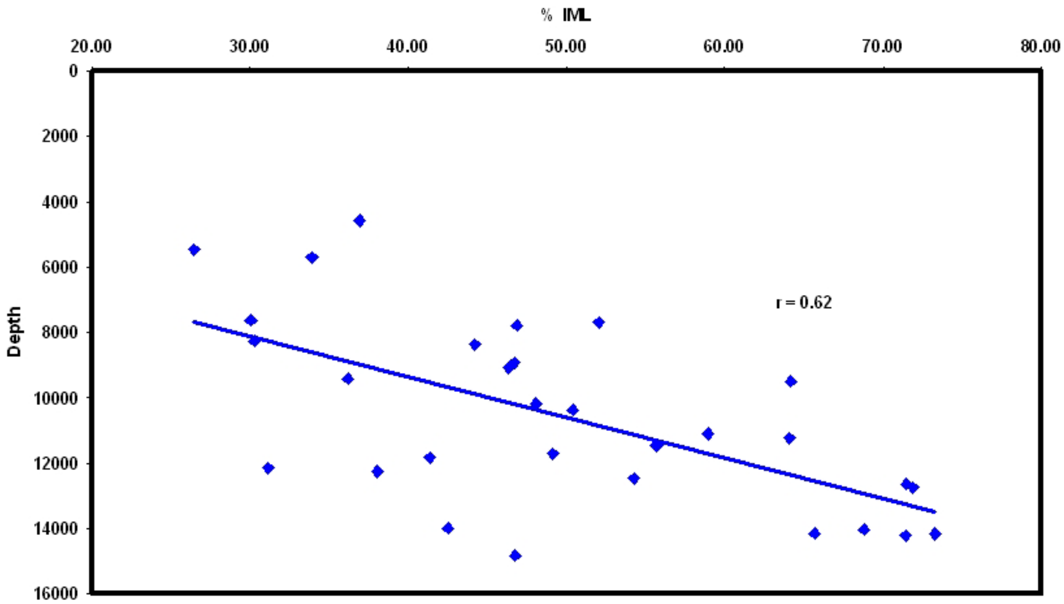

Figure 14: Multiple IML samples correlates with depth (r=0.62) at the expense of smectite

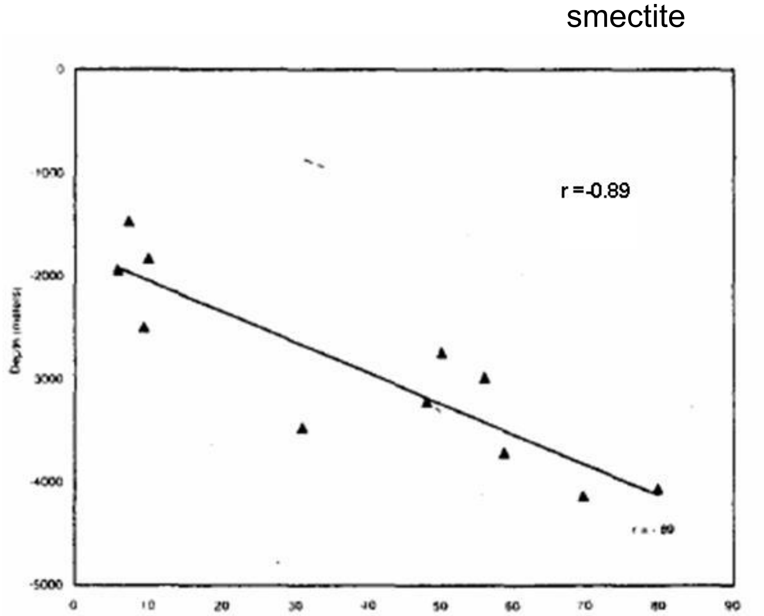

Figure 15: IML correlation with Depth (r =-0.89 from the single well study in Ship Shoal (Totten et al, 2002)

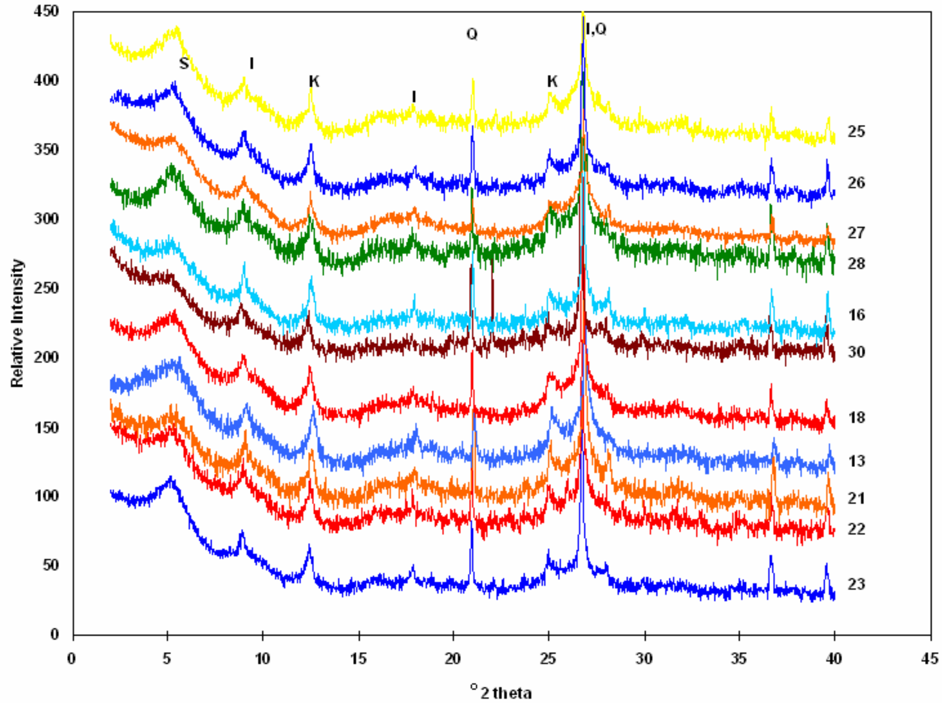

Figure 16: IML increases during diagenesis and exhibits a consistent mineralogy in each sample. The IML fraction is consistently an illite dominated mixed layer clay. Each diffraction pattern corresponds to the sample number listed on the right.

Smectite

Distinct differences are apparent in both the end-member smectite (EMS) and the smectite-rich mixed-layer (SML) fractions comparing the Ship Shoal single well study and this multi-well study. This study has a larger variation in the EMS component. Seven samples in this study contained over 15% of the EMS fraction, while the same fraction in the Ship Shoal single well study averaged 5 %, and had a maximum value in the shallowest sample at 12%.

Examination of the EMS fraction in this study shows two different populations. The first set of samples has a low percentage of the EMS fraction, and is consistent with the results described in the Ship Shoal single well study (Totten et al. 2002). The second set has a significantly higher amount of EMS, and shows a very different character as seen by the X-Ray diffractograms. Figure 17 shows low percentage EMS XRD patterns, which are comparable to the XRD results in the Ship Shoal study (Figure 18). Figure 19 illustrates the XRD patterns for samples with higher EMS percentages. The difference is an increase in intensity of the kaolinite peak, which suggests a mixed-layer kaolinite smectite clay that has a lower density than a mixed layer illite smectite clay, based on theoretically derived densities. Weaver (1989) reports significant randomly interstratified kaolinite/smectite from Georgia and the Coastal Plain of the United States, and suggests that they are probably more abundant than generally realized. A significant increase in this component within the EMS would explain the increased percentage of this fraction compared to the samples without significant K/S.

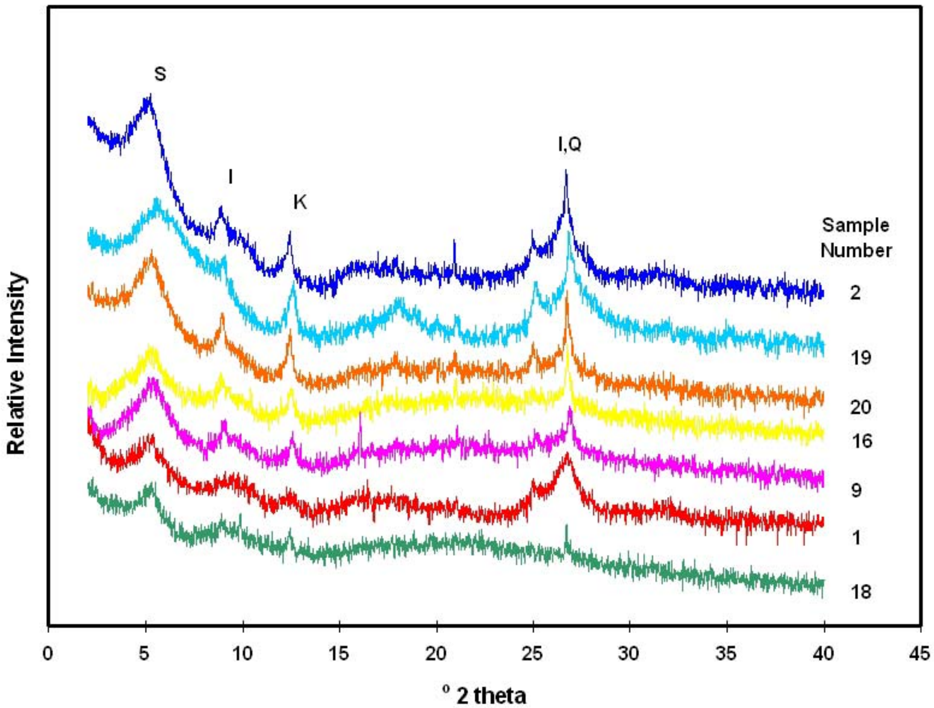

Figure 17: This figure shows a low percentage of EMS in this study. These samples are similar to the results that were published from a Ship Shoal single well study (Totten et al. 2002).

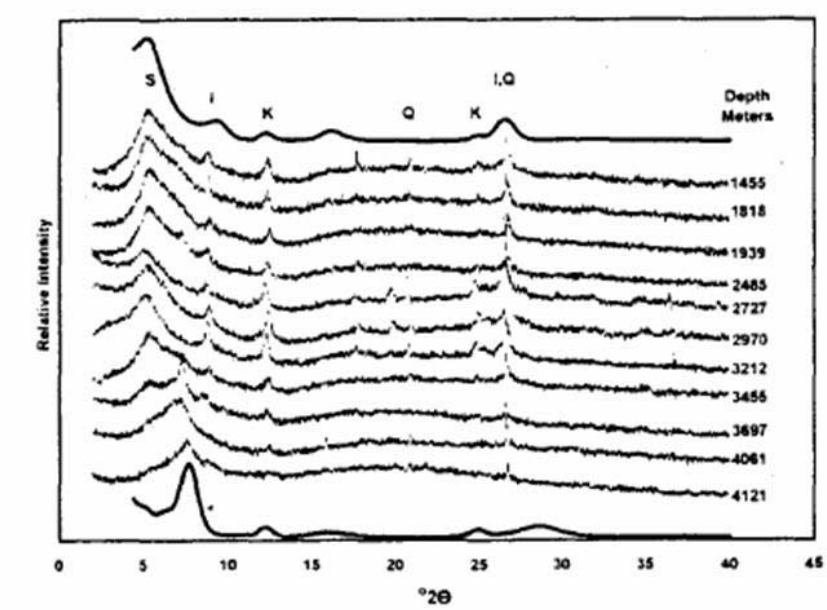

Figure 18: Low percentage EMS in the single well Ship Shoal study (Totten et al, 2002)

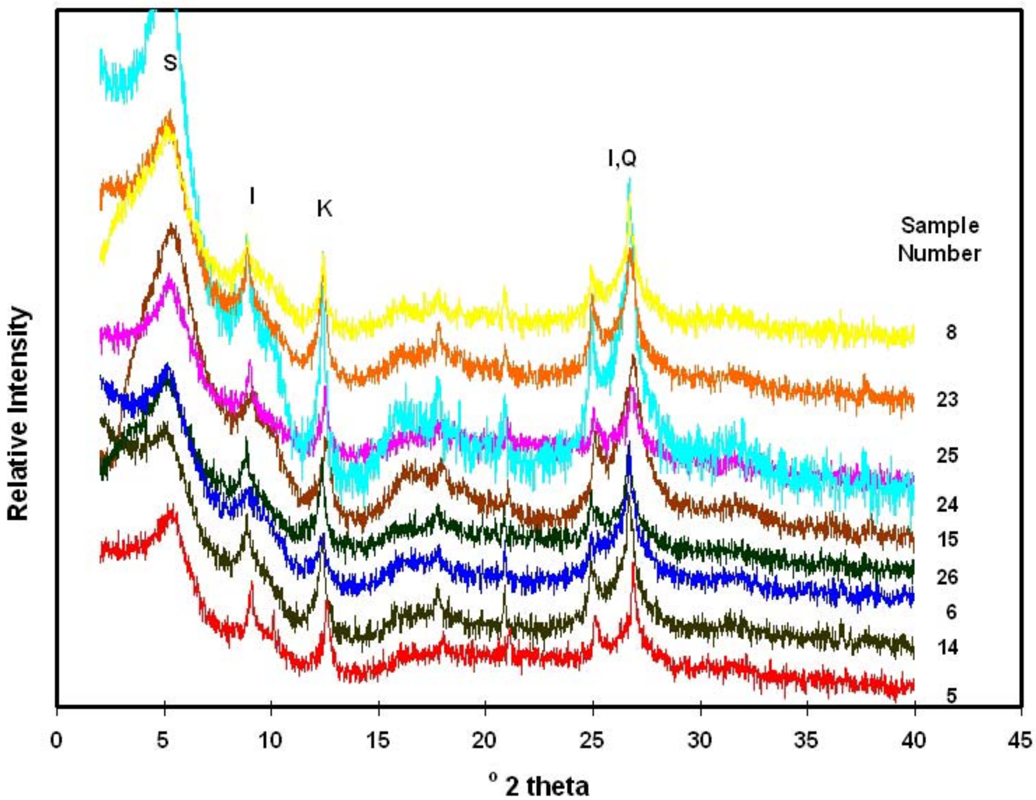

Figure 19: This figure shows the XRD patterns for the samples with higher EMS percentages. Note the increased intensity of the kaolinite peak, which may suggest a mixed layer kaolinite – smectite clay.

The large EMS component variability may also explain the unpredictable swelling behavior observed during drilling operations in this area. This component is not observed by XRD of the entire clay fraction, but is exposed in some of the lighter fractions after density separation using LMT. The kaolinite peak (2 theta =12.4°) is evident in the EMS and SML fractions as compared to the IML, EMI, and the non-separated sample shown in Figure 13, and also note that the kaolinite peak is inconspicuous in the bulk, non-separated sample**,** yet this sample contained 59% of the smectite/kaolinite-rich fractions (EMS & SML).

The smectite rich components (EMS and SML) do not correlate with depth or temperature. The SML percentage is variable and has a conspicuous kaolinite component when compared to the EMS fraction. When EMS and SML components are combined, they decrease with depth, in an inverse relation to the illite-rich clay component.

Conclusions

Generally speaking, the trend of the clay minerals in the shales collected in South Timbalier for this multi-well study is toward an increased illite-rich mixed-layer component with increased depth, consistent with many previous studies. The clays do not correlate well with current, modern bottom hole temperatures. Although this multi-well study demonstrates that there is a correlation with illitization and depth, it is not as strong as the single well study in Ship Shoal. Clay-mineral diagenesis is a complex reaction dependent on many variables. It is likely that single well studies limit many of these variables, and may exaggerate the apparent control of depth on clay-mineral reactions.

An interesting result of this study is the variability of the smectite-rich clays. This result is not obvious using standard clay mineral XRD techniques, but is apparent using the separation method outlined in this study. The large variation in these expandable clays could explain the unpredictable behavior of these rocks during drilling operations.

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Vita

Mark Dallas Dixon was born in San Angelo, Texas January 30, 1966. As a young child, he lived in Monahans, Texas until 1974, when he moved with his family to Western Wyoming until 1978. His family returned to Texas and settled in San Angelo, where he finished junior high and high school. He received a bachelor of science degree in the field of Geology from Angelo State University in December 1990, the last graduate from the tiny department. He is currently living in New Orleans and is employed with Halliburton Energy Services as a technical professional.