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Diffusion Osmosis-An Unrecognized Cause of Shale Instability

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Abstract

Problems encountered while drilling shale formations are a major factor in the cost of oil and gas wells. A principal cause of the problems has been shown to be the transfer of water and ions from water-based drilling fluids to shale formations. Prior studies have documented two driving forces involved in such transfer. One is the hydraulic pressure differential between the drilling fluid and shale pore fluid. A second is a chemical osmotic force dependent upon the difference between the water activity (vapor pressure) of the drilling fluid and that of the shale pore fluid under downhole conditions. Generally unrecognized is another driving force, diffusion osmosis, which is determined by the difference in concentrations of the solutes in the drilling fluid and shale pore fluid. Diffusion osmosis results in transfer of solutes and associated water from higher to lower concentration for each species, opposite to the flow of water in chemical osmosis. If the diffusion osmotic force exceeds the chemical osmotic force, invasion of ions and water can increase the pore pressure and water content of the shale near the borehole surface. Additionally, the invading ions can cause cation reactions that alter the clay structure in the shale. All of these effects tend to destabilize the shale.

Destabilizing ionic reactions within a shale can be minimized if a suitable nonionic polyol (such as methyl glucoside) is used to reduce the activity of a fresh-water drilling fluid. In certain situations the addition of salt to such a fresh-water drilling fluid to obtain further reduction of water activity can cause an increase in the diffusion osmotic force that offsets some, or all, of the desired increase in chemical osmotic force. This now is recognized to have probably been

a factor when sodium chloride was included in the formulation of a methyl glucoside drilling fluid used with moderate success for drilling in the Gulf of Mexico.

Chemical osmotic effectiveness can be improved by emulsification of a non-aqueous phase in the drilling fluid. A fresh-water drilling fluid containing methyl glucoside for activity control and emulsified pentaerythritol oleate prevented hydration and maintained stability of Pleistocene shale from the Gulf of Mexico. Drill cuttings from such a drilling fluid should be environmentally acceptable for discharge at offshore or land locations.

Introduction

Interactions of water-based drilling and completion fluids with shale formations have long been recognized as a major factor in the cost of finding and producing oil and gas. Much progress has been made in understanding the mechanisms responsible for the destabilization of shale and subsequent problems such as high torque, stuck pipe, lost circulation and cementing failures. In most instances the problems can be avoided by use of hydrocarbon-based drilling fluids, but the use of those fluids are now being curtailed because of environmental concerns. The excellent shale stability provided by hydrocarbon-based fluids has been attributed in part to the establishment of an essentially ideal semipermeable membrane at the drilling fluid/shale interface. The membrane has openings large enough to allow water molecules to pass, but small enough to prevent passage of dissolved ions and molecules. This results in development of an osmotic pressure differential which is dependent upon the ratio of water activity (escaping tendency) of the drilling fluid (a_{df}) to that of the shale pore fluid (a_s). The transfer of water is from the higher water activity (lower concentration of dissolved ions or molecules) to the lower water activity. Solutes, such as calcium chloride, can therefore be added to the water phase of a hydrocarbon-based emulsion fluid to reduce the water activity and develop enough osmotic force to prevent shale hydration, or even extract water from shale. This mechanism for water transfer is commonly designated chemical osmosis.

In recent years it has been postulated that water-based fluids form a non-ideal semipermeable (leaky) membrane at a shale interface. $^{2,\ 3}$ In addition to transfer of water, limited

transfer of solutes also occurs. This driving force generally has been considered as chemical osmosis, but with a membrane efficiency factor to account for the lesser osmotic force observed.

Major progress in understanding drilling fluid/shale interactions has been made since the development of equipment and procedures that permit study of those interactions under close approximation of downhole conditions. The experiments use specimens of preserved downhole that was cored using a hydrocarbon-based mud. After restoration to in-situ stresses and temperature, the shale specimen is drilled and exposed to the drilling fluid to be tested.⁴ These procedures avoid artifacts of commonly used testing methods such as air in exposed shale pore spaces or flushing of shale with an arbitrary simulated pore fluid prior to exposure to the drilling fluid to be tested. This Downhole Simulation Cell (DSC) equipment in the OGS Laboratory in Houston, Texas, has been utilized in studies funded by Gas Research Institute (GRI) to test Cretaceous shale cores from the U.K. sector the North Sea.^{5, 6} Some of the conclusions from those studies were:

- The hydraulic differential between the borehole pressure and the far-field shale pore pressure was a driving force affecting the transfer of water between either a hydrocarbon-based emulsion or a water-based drilling fluid and a low-permeability shale. The hydraulic force could either support or oppose an osmotic force.
- Osmotic pressure was a driving force affecting the transfer of water between a drilling fluid and a lowpermeability shale. The water activity of either a hydrocarbon-based emulsion or a water-based drilling fluid could be adjusted to cause water to enter or be extracted from a shale.
- Even if water was extracted from a shale osmotically by use of a brine having a very low water activity, ions penetrated the shale and caused a reduction in borehole stability.

The most recent DSC studies of drilling fluid/shale interactions funded by GRI⁷ have been made using preserved cores of Pleistocene shale from the Gulf of Mexico. This shale is younger and less compacted than the Cretaceous shale previously tested. During the course of the recent program, Harold W. Olsen, an advisor to the project, suggested that sufficient attention had not been given to a distinction in the osmotic forces involved in the transfer of water between water-based fluids and shales. In his paper, "Chemico-Osmosis Versus Diffusion-Osmosis," Olsen stated that a concentration gradient in soil can cause convection, or drag, of bulk pore fluid by diffusion of the solute species from concentrated to dilute solutions. This diffusion osmosis opposes chemical osmosis, which causes movement of water from dilute solutions (high water activity) to more concentrated solutions (lower water activity). He further stated that diffusion osmosis and chemical osmosis progress simultaneously. Chemical osmosis is most effective in densely compacted formations exhibiting high cation exchange capacity while diffusion osmosis is favored by less compaction and lower cation exchange capacity.

When diffusion osmosis as described by Olsen is considered, the interactions of water-based drilling fluids and shale formations become even more complex because each solute species in both the drilling fluid and shale pore fluid becomes a factor. The transfer of solute and water depend not only upon the relative concentration of each solute in the drilling fluid and pore fluid, but also upon the solute selectivity of the drilling fluid/shale interface at the downhole conditions.

With the Gulf of Mexico Pleistocene shale being far less compacted than the North Sea Cretaceous shale previously studied, the recent DSC tests presented an opportunity to observe for and determine the importance of diffusion osmotic effects. Unlike the work cited by Olsen, specimens of the preserved shale core could be restored to downhole conditions and drilled with no need for flushing with a simulated pore fluid.

Pleistocene Shale Cores from the Gulf of Mexico

The cores for this project were obtained by Texaco Exploration & Production, at the request of their Technology Department, from their OCS-G-2937 Well A-42 in West Delta Block 49 of the Gulf of Mexico. Water depth at the location was 208 ft and casing was set at 3,835 ft. A 9.8-lb/gal synthetic-based mud was used to cut 4-inch core from 4,128 to 4,215 ft. The downhole conditions at the average core depth were estimated to be as follows:

Drilling Fluid Pressure 2,200 psi Temperature 150°F Vertical Stress 3,450 psi Horizontal Stress 2,650 psi Pore Pressure 2,000 psi

The core specimens were later restored to these conditions when DSC tests were made. The DSC test procedures used are described in Appendix A.

A technologist from the OGS Laboratory was at the well site to coordinate the preservation of the shale core. The plastic core sleeves were cut into 3-foot sections, immediately sealed with rubber end caps and placed in wooden boxes identified as to depth. The boxes were brought to shore and taken by truck to the OGS Laboratory. There the cores were carefully removed from the sleeves, wiped and blotted to remove as much mud as practical, and then wrapped in plastic film. To lessen breakage, the pieces of core were wrapped with duct tape prior to being sealed by painting with beeswax. The cores were then returned to the identifying wooden boxes. Since an electrohygrometer test had indicated the water activity of the shale to be about 0.89, the boxes were stored in a sealed area where the humidity was maintained at about 89%.

Texas E&P Technology Department provided some very helpful mercury injection capillary pressure test results comparing the Pleistocene shale used in this investigation with the Cretaceous shale previously studied in this GRI project. Based on their data, the Gulf of Mexico Pleistocene core might best be described as a mudstone as compared to the more compacted North Sea Cretaceous shale.9 However, in this paper both cores are considered as being within the broad classification of shales. As shown in **Table 1**, the porosity of the Pleistocene shale was about twice that of the Cretaceous, but the calculated air permeability was about 75 times as great. The mean pore aperture diameter of the Pleistocene was in the order of 0.08 micron as compared to only 0.009 micron for the With much larger pore sizes and higher Cretaceous. permeability, the Pleistocene shale would be expected to be far more susceptible to diffusion osmotic effects than the Cretaceous shale previously studied.

Water Activity of Shale at Downhole Conditions

In prior studies of the Cretaceous shale from the North Sea, the use of an electrohygrometer to test the humidity above pieces of the preserved shale indicated a water activity of about 0.78 at ambient conditions. It was assumed that an oilbased emulsion drilling fluid providing an ideal semipermeable membrane at the shale surface would result in no osmotic pressure if the activity of the drilling fluid were adjusted to 0.78. However, a DSC test of an oil-based emulsion drilling fluid having that ambient activity resulted in fluid being extracted from the shale at a substantial rate when there was no hydraulic differential pressure. It was concluded that the activity of the shale at downhole conditions was higher than 0.78. Plans were made to test oil-based emulsion fluids having higher activities to determine that needed to balance the activity of the shale, but the supply of shale core was exhausted before such tests could be made.

In the study of the Pleistocene shale, priority was given to the determination of drilling fluid activity at ambient conditions to provide balancing activity for the shale at downhole conditions. The electrohygrometer test of pieces of preserved Pleistocene shale indicated the ambient activity to be 0.89. An oil-based emulsion drilling fluid was prepared from mineral oil and calcium chloride brine to obtain an ambient activity of 0.89. (See Appendix B for drilling fluid compositions.) A DSC test of this drilling fluid was made with the borehole pressure equal to the initial pore pressure of the shale (no hydraulic pressure differential). After an equilibration period, exposure to the drilling fluid resulted in fluid being extracted from the shale at a very slow steady rate of 0.06 ml/hr. The fluid transfer rate was not quantitative because passage of oil from the sandpack to the outer periphery of the shale specimen was hindered by capillary forces. The shale specimen was in excellent condition after exposure to the drilling fluid as can be seen in Fig. 1. There was no substantial change in moisture content of the shale, but the penetrometer hardness near the borehole surface was considerably lower than before the DSC test (Table 2). This might have been caused by the specimen being subjected to elevated pressure and temperature and then returned to ambient conditions. Exposure to the drilling fluid caused no significant change in exchangeable bases (**Table 3**), indicating that the oil-based emulsion fluid provided a very efficient semipermeable membrane that prevented diffusion of ions.

Similar results were obtained when an oil-based emulsion fluid having an ambient activity of 0.93 was tested. However, an oil-based emulsion drilling fluid having an ambient activity of 0.97 caused fluid transfer into the shale at a very slow rate of 0.04 ml/hr. It was concluded from these tests that a drilling fluid having a water activity of about 0.95 at ambient conditions would balance the water activity of the Pleistocene shale at downhole conditions.

DSC Test of Water-Based Fluid at Balanced Activity

A simulated shale pore fluid to be used as a drilling fluid was prepared to have an ambient activity of 0.95, the activity indicated to balance the activity of the shale at downhole conditions. The cations for the simulated pore fluid were in the same ratios as measured for the exchangeable bases of the shale. Chloride salts of these cations were used to assure solubility over the temperature range of ambient to 150°F. A DSC test was made using the simulated pore fluid as the drilling fluid. In spite of the borehole pressure and initial shale pore pressure being equal (no hydraulic pressure differential) and the drilling fluid having a water activity to balance that of the shale at downhole conditions (no chemical osmotic differential), the DSC test showed fluid transfer from the drilling fluid to the shale at a high rate of 0.78 ml/hr. Decreases in axial stress showed axial compaction of the shale specimen after about 3 hours of drilling fluid circulation and the borehole collapsed after 7 hours. (See photo in Fig. 1.) Subsequent examination of the shale showed the moisture content had increased and the penetrometer hardness had been greatly reduced throughout the specimen. The analyses of exchangeable bases showed some reduction in sodium, but increase in other cations near the borehole surface (Table 3). The results of this DSC test demonstrate the importance of diffusion osmosis as a driving force in the transfer of solutes and associated water between drilling fluid and shale. With no chemical osmotic differential and no hydraulic pressure differential, the diffusion osmotic force caused invasion of water and ions that weakened the shale and caused borehole collapse within a few hours of drilling fluid exposure.

Diffusion Osmotic Mechanisms

Attention to the ion analyses of the shale and drilling fluid used in the above DSC test can provide insight into the mechanisms involved in diffusion osmosis. Shown in **Table 4** are the ion concentrations found in the Pleistocene shale pore fluid and in the simulated pore fluid used as a drilling fluid for the DSC test. The first column lists the exchangeable cations in milliequivalents per 100g of dry shale. These cations are balanced by negative charges on the clays in the shale; i.e., the clays are the anions. The second column lists the

concentrations of the cations and anions that were analyzed in an alcoholic water extract of the shale. The third column lists the total of the cations in the exchange sites of the shale plus the extractable cations and anions in terms of millequivalents in the water associated with 100g of dry shale. The fourth column lists the total ion concentrations as milliequivalents per liter of shale pore water. The fifth column lists the concentrations of ions in the simulated pore fluid used as the drilling fluid.

Obviously, the drilling fluid composition does not truly simulate the pore fluid. In formulating the drilling fluid, chloride salts were used intentionally. Had carbonate and sulfate been used, calcium would have been precipitated. Of greater significance, the total concentration of soluble anions in the shale pore fluid (400 meq/L) is far less than the concentration of anions in the drilling fluid (1600 meg/L). This anion deficiency of the shale pore fluid is caused by the clays in the shale acting as multivalent insoluble anions. For this reason, the shale pore fluid cannot exist outside of the shale and cannot be truly simulated with soluble salts. The drilling fluid, having the desired activity of 0.95 and cations in the same ratios as the exchangeable cations in the shale, must have soluble anions equivalent to the cations. consequence the excess of anions in the drilling fluid will develop a diffusion osmotic force that drives anions, as well as associated cations and water, into the shale.

The data in Table 4 show the total sodium concentration of the shale pore fluid to be substantially greater than the sodium in the drilling fluid, indicating that diffusion might cause sodium to be transferred from the shale to the drilling fluid. The analyses of exchangeable bases after the shale was exposed to the simulated pore fluid as a drilling fluid did in fact show a reduction in sodium, while there was an increase in other cations in the exchange sites of the shale specimen near the borehole surface.

Diffusion osmosis can be expected to occur during ordinary drilling operations utilizing water-based drilling fluids. There will be an imbalance between specific cations and anions in the shale pore fluid and the drilling fluid, and diffusion osmosis will cause a corresponding transfer of cations and anions. The net flow of water can be either into or out of the shale, depending upon the resultant of diffusion osmosis, chemical osmosis and hydraulic pressure differential. The role of the membrane established at the drilling fluid/formation interface is illustrated in Fig. 3 for the condition of drilling fluid pressure equal to the far-field pressure. For example, for water-based drilling fluid in a permeable sand there would be no membrane and no osmosis. Water would diffuse between the drilling fluid and sand from high to low water activity. Each ionic species would diffuse from high to low concentration, probably with some species entering and some leaving the sand. There would be no change in the sand formation pore pressure. In contrast, a hydrocarbon-based emulsion drilling fluid would provide an ideal membrane at a shale interface. There would be no diffusion, and water transfer would be controlled solely by chemical osmosis. Water-based drilling fluid would provide a non-ideal semipermeable membrane at a shale interface. There would be both chemical and diffusion osmosis, with all of the associated complexities.

The schematic in Fig. 3 deals with drilling fluids containing only inorganic solutes. In actual field operations, the interactions between drilling fluids and shale formations can become even more complex when organic solutes that alter water activity are incorporated in water-based drilling fluids. An example of this was provided by a prior DSC test⁴ in which glycol was added to a fresh-water drilling fluid to obtain an ambient water activity considerably lower than that of a North Sea Oligocene shale. The glycerol diffused into the shale, expanded the clay structures, and caused more transfer of fluid into the shale than had been observed for a drilling fluid using potassium chloride to obtain the same water activity as the glycol system. In another study,⁵ a DSC test of North Sea Cretaceous shale was made using potassium formate brine having a water activity far lower than that of the shale. The chemical osmotic force developed at the interface with the highly compacted shale was great enough to extract fluid from the shale in spite of the opposing diffusion osmosis caused by the high concentration of potassium ions in the drilling fluid. However, the invading potassium replaced other cations in the exchange sites of the clays in the shale, altered the fabric of the clays and reduced the relative stability of the shale. In contrast, the use of nonionic methyl glucoside to lower the activities of fresh-water drilling fluids to less than the activities of the Oligocene and Cretaceous shales caused no detrimental diffusion effects in DSC tests.^{4,5}

DSC Tests of Calcium Chloride to Obtain Water-Based Fluids with Low Water Activity

Use of a simulated pore fluid having an ambient activity of 0.95 as the drilling fluid failed to prevent transfer of fluid from the drilling fluid to the Pleistocene shale, even when there was no hydraulic pressure differential. Since the ambient activity of the drilling fluid was equal to that found to balance the activity of the shale at downhole conditions, the fluid transfer was attributed to a diffusion osmotic force in the absence of an opposing chemical osmotic force. It was therefore decided to test a drilling fluid containing a higher concentration of ions to obtain a lower ambient water activity and a chemical osmotic force that might extract fluid from the shale.

A DSC test was made using a drilling fluid composed of 10.3-lb/gal calcium chloride brine having an ambient activity of 0.72. In spite of the low ambient activity of the drilling fluid, fluid was transferred into the shale. The rate was about one-third that observed when simulated pore fluid having an activity of 0.95 was used as the drilling fluid, but compaction of the shale specimen was noted after about three hours of circulation and the specimen collapsed after about 12 hours. Subsequent examination of the specimen revealed that the

moisture content near the borehole had increased and the penetrometer hardness was zero (Table 2). Although there was no increase in moisture farther into the shale, calcium was found to have replaced sodium in the exchange sites of the shale as far as 1.5 inches from the borehole. These test results indicated that the diffusion osmotic force developed by the relatively high concentration of calcium chloride in the drilling fluid was greater than the chemical osmotic force developed by the reduced drilling fluid activity.

Fig. 4 shows the fluid transferred at the sandpack for this test compared to the results of the previously discussed DSC test using the oil-based emulsion drilling fluid having an ambient water activity of 0.89. The mineral oil emulsion with its ideal semi-permeable membrane extracted water from the shale after an initial equilibration period. This can be compared to the transfer of fluid to the shale by the calcium chloride brine having an ambient activity of 0.72. The comparison shows that the flow into the shale occurs with a fluid that has a substantially lower activity than that of the shale. Diffusion osmosis dominating chemical osmosis offers an explanation of why this occurs.

Another DSC test was made using an 11-lb/gal calcium chloride brine drilling fluid having an even lower ambient activity of 0.54. This drilling fluid did result in fluid being extracted from the shale at a very low rate. There was no indication of compaction or failure of the shale specimen during the circulation period. However, examination of the specimen after removal from the DSC revealed a band of concentric failure rings around the borehole that was about 1/4 inch in thickness. (See photo in Fig. 1.) The penetrometer hardness of the shale near the borehole surface was zero. The hardness readings beyond that band did not indicate failure or softening. Although there was no substantial change in moisture content of the shale, the analyses of exchangeable bases showed that calcium had replaced sodium as far as 1.5 inches from the borehole.

The results of this test were both surprising and significant. There appears to have been a diffusion osmotic force that transported hydrated calcium ions into the shale while a greater chemical osmotic force caused a net transfer of fluid from the shale to the drilling fluid. Cation exchange of calcium for sodium in the exchange sites of the clavs in the shale probably altered the fabric of the shale and caused the band of concentric failure rings. This band could be observed because the DSC test conditions provided laminar flow in the annulus between the static simulated drill pipe and the borehole wall. In actual drilling operations the failed shale at the borehole wall would be knocked off by drill string rotation and tripping, causing hole enlargement, loss of annular velocity, and poor hole cleaning. Those circumstances would then lead to typical shale problems of high torque, drag, fill, stuck pipe and lost circulation.

DSC Tests of Nonionic Polyol to Reduce Drilling Fluid Activity

The tests of calcium chloride brines to drill the Pleistocene shale demonstrate the basic problems associated with attempts to utilize ionic solutions to prevent shale hydration and weakening. The concentrations of ions required to reduce the activity of the drilling fluid to obtain chemical osmotic force to extract water from the shale also develop diffusion osmotic forces that drive ions into the shale while opposing extraction of water. The ions entering the shale can have destabilizing effects even if water is extracted.

DSC tests of the Speeton Cretaceous shale⁵ had shown that ionic effects could be minimized by use of an environmentally acceptable nonionic solute (methyl glucoside) in fresh water to lower the activity of the drilling fluid and provide a more efficient chemical osmotic membrane at the shale surface. A methyl glucoside solution having an activity of 0.78 extracted fluid from the shale and maintained shale stability in spite of a hydraulic pressure differential of 400 psi tending to drive fluid into the shale. The performance was quite similar to that of an oil-based emulsion drilling fluid having an activity of 0.78

The Gulf of Mexico Pleistocene shale, having a higher porosity and permeability than the Speeton shale, would be expected to make the development of an efficient chemical osmotic membrane more difficult. In order to determine if a solution of methyl glucoside in fresh water could develop enough chemical osmotic pressure to extract fluid from the shale, a DSC test was made using 70% w/w methyl glucoside in fresh water as the drilling fluid and with zero hydraulic pressure differential. This drilling fluid (water activity of 0.78) resulted in fluid being extracted from the shale at a very low rate of 0.02 ml/hour, indicating a chemical osmotic force that exceeded any diffusion osmotic force. As shown in Fig. 1, the exposed shale specimen was in good condition. The analyses of exchangeable bases (Table 3) indicated some minor replacement of other cations by potassium from the potassium hydroxide used to neutralize the MEG 207 methyl glucoside product and adjust the drilling fluid pH to 9.5. Exposure to the drilling fluid caused no significant change in moisture content of the shale. Penetrometer hardness readings were lower after exposure, but were similar to those measured in prior tests of oil-based emulsion drilling fluids that extracted fluid from the Gulf of Mexico shale. (See Table 2.)

The methyl glucoside solution in the above test served to extract water from the Gulf of Mexico shale and maintain shale stability when the hydraulic pressure differential was zero. However, in most drilling operations borehole pressure is maintained higher than the formation pore pressure. Therefore, a DSC test was made to observe the effect of a 200-psi hydraulic differential pressure when using a 70% w/w methyl glucoside solution. This DSC test showed fluid being transferred from the drilling fluid to the shale at a low rate, and there was loss of borehole stability as indicated by the

photo in **Fig. 2**. Examination of the shale specimen showed it to be very similar to that of the previous test as to exchangeable bases, moisture and hardness. The two DSC tests showed that the net osmotic pressure developed by the 70% w/w methyl glucoside was less than 200 psi.

Effect of an Emulsified Non-Aqueous Liquid Phase

It was decided to investigate improving the effectiveness of the osmotic membrane provided by the 70% methyl glucoside solution by including a dispersed non-aqueous liquid phase. A commercially available ester lubricant (pentaerythritol oleate) was selected. Lignite was included in the drilling fluid formulation as a simple means of providing additional emulsion film strength. A DSC test was made using a drilling fluid having an ambient water activity of 0.80 and containing 0.9 bbl of a 70% w/w methyl glucoside solution, 0.1 bbl of ester lubricant, 10 lb of lignite and 2 lb of potassium hydroxide per bbl of fluid. The hydraulic pressure differential was 200 psi. The DSC test showed that fluid was extracted from the shale to the drilling fluid at a slow steady rate of about 0.03 ml/hr. Examination showed the exposed shale specimen to be in excellent condition, with the borehole smooth and gauge. (See photo in Fig. 2.) There was no substantial change in moisture content of the shale specimen. The penetrometer hardness readings throughout the specimen were higher than the values measured for the oil-based emulsion fluids (Table 2). There was no change in exchangeable bases other than a slight increase in potassium near the borehole surface. The properties of the drilling fluid were quite stable during the DSC test. The rheological properties were very high at 75°F, but were reasonable at 120°F. The API filtrates were zero and the filtrates at 200°F were extremely low, both during and after the circulation period.

The condition of the shale specimen exposed to this drilling fluid was as good as, or better than, specimens exposed to the oil-base emulsion fluids. performance concern would be the high viscosity when tested at 75°F. Like some oil-based or synthetic-based drilling fluids, methyl glucoside solution is much more viscous at temperatures below 100°F. It was decided to investigate whether good shale stability could be obtained using a similar drilling fluid prepared from methyl glucoside diluted to 60% w/w with sodium chloride brine to provide lower viscosity while maintaining about the same water activity of 0.80. A DSC test was made using a drilling fluid composed of about 0.77 bbl of 70% w/w methyl glucoside solution, 0.13 bbl of 10-lb/gal sodium chloride brine, 0.1 bbl of ester lubricant, 5 lb of starch per bbl of fluid. Potassium hydroxide was added to obtain a pH of 10. The hydraulic pressure differential was 200 psi. The DSC test showed that fluid was extracted from the shale to the drilling fluid at a very slow rate of about 0.01 ml/hr. Examination of the shale specimen after removal from the DSC showed the borehole to be essentially gauge and in fairly good condition. There was no substantial change in moisture content, but there was some reduction in hardness near the borehole surface (Table 2). Analysis of the exchangeable bases showed some increase in sodium and potassium and reduction in calcium throughout the shale specimen. The drilling fluid had good rheology but poor filtration control at high temperature.

The fresh-water drilling fluid containing 70% w/w methyl glucoside, an ester lubricant and lignite to obtain water activity of 0.80 had resulted in performance as good or better than that of an oil-base emulsion having an activity of 0.93, except that the rheological properties at 75°F were too high for most drilling operations. Therefore, it was decided to test the same formulation, except use fresh water to dilute the methyl glucoside concentration to 60% w/w to obtain lower viscosity. The resulting drilling fluid had an activity of 0.88. The hydraulic pressure differential for the DSC test was 200 psi. The DSC showed that fluid was extracted from the shale to the drilling fluid at a rate of 0.01 ml/hr, the same as observed in the previous DSC test using the drilling fluid having a water activity of 0.80. (See Table 2.) Examination of the shale specimen after removal from the DSC showed the borehole to be in gauge and in very good condition. There was no substantial change in moisture content. The reduction in shale hardness near the borehole surface was the same as observed in tests of the oil-based emulsion fluids. Analyses of the exchangeable bases showed only a small increase in potassium and decrease in sodium near the borehole surface. The rheological properties were satisfactory for most drilling operations. The API filtrates were very low. While the filtration rate at 200°F was high initially, circulation reduced it to very low value by the end of the test.

Discussion of Effects of Salt on Nonionic Polyol Drilling Fluid

Comparison of the DSC tests of the two drilling fluids containing 60% w/w methyl glucoside indicated no significant advantage to the inclusion of sodium chloride to obtain an activity of 0.80 instead of 0.88. The increase in diffusion osmotic force tending to drive solutes and associated water into the shale fully offset the anticipated increase in chemical osmotic force tending to extract fluid from the shale. A disadvantage in certain operations would be that inclusion of salt could preclude on-site disposal of drilling fluid and cuttings. The results of the two DSC tests were significant for another reason. They showed that help in avoiding shale problems could still be provided by a methyl glucoside drilling fluid even if salt became incorporated from salt water flows or the drilling of salt formations.

From the perspective of field operations, it is important to know that the addition of soluble salts to water-based drilling fluids to reduce water activity also causes diffusion osmotic forces that can offset some or all of the desired increase in chemical osmotic force. The significance of these opposing osmotic forces can be illustrated by consideration of the first uses of methyl glucoside fluids for drilling in Pleistocene shale in the Garden Banks area of the Gulf of Mexico. 10 Since there

was no Gulf of Mexico Pleistocene shale core available at that time, the drilling fluid composition for these wells was selected based on DSC tests of preserved Oligocene shale core from the Danish sector of the North Sea. Those tests showed that shale hydration and instability could be avoided by use of a fresh-water drilling fluid containing 44% w/w methyl glucoside to obtain a water activity of 0.88. However, similar results were obtained using a fluid containing only 25% w/w methyl glucoside in conjunction with 115,000 mg/L of sodium chloride to provide a water activity of 0.86. The latter composition was chosen for the drilling operations because of the substantially lower cost per barrel. Various logistical problems resulted in the two wells being drilled with drilling fluid containing only about 15% w/w methyl glucoside. In spite of the low concentration of methyl glucoside, the drilling conditions and costs were better than for most of the other wells drilled from the platform. Nonetheless, there was occasional bottom hole assembly balling and pumping out on short trips, indicating some shale hydration and instability.

In retrospect it can be recognized that the DSC tests of the Oligocene shale did not indicate the diffusion osmosis that probably opposed chemical osmosis in the Garden Banks Pleistocene shale. The Oligocene shale core had a moisture content of 15% and a water activity of 0.91. These values were fairly similar to the moisture content of about 12% and water activity of 0.89 determined for the West Delta Pleistocene shale core obtained later. Unknown at the time, however, was that other properties of the North Sea Oligocene shale were quite different from those of the Pleistocene shale from the Gulf of Mexico. For example, the cation exchange capacity of the Oligocene shale was 32.3 meq/100g, as compared to 17.2 for the Pleistocene shale. Perhaps of even greater importance, the mean pore aperture diameter of the Oligocene shale was 0.032 micron as compared to 0.076 micron for the Pleistocene. Based on these latter two parameters, the Oligocene shale would favor chemical osmosis while the Pleistocene shale would be more subject to diffusion osmosis. Had the Pleistocene shale core been available prior to the Garden Banks drilling operations, DSC tests could have warned against inclusion of the salt and shown the advantage of obtaining the desired activity with methyl glucoside in the 44% w/w concentration range. DSC tests such as those presented in this paper would also have shown the advantage of emulsifying a suitable nonaqueous phase.

Future Research Needs

Additional research is needed on methods of maximizing chemical osmosis to extract fluid from shale into a drilling fluid while minimizing diffusion of reactive solutes into the shale. These studies could be two fold:

 Investigation of materials to reduce water activity of drilling fluids with minimal increase in diffusion osmosis, drilling fluid viscosity and cost.

- 2. Investigation of drilling fluid materials to reduce shale permeability and improve chemical osmotic effectiveness. Examples would be:
 - An emulsified non-aqueous phase.
 - Water-dispersible material for adsorption and plugging within the shale.
 - Water-soluble material for precipitation within the shale.

Conclusions

Diffusion osmosis is a driving force affecting the transfer of solutes and associated water between water-based drilling fluids and shale formations. The diffusion osmotic force is determined by the differences in the concentrations of the individual solutes in the drilling fluid and shale, and the flow is from high to low concentration. Such flow is affected by the solute selectivity of the drilling fluid/shale interface at downhole conditions for each individual solute (either ions or molecules) in contact with that specific shale. Invasion of water and solutes weaken and tend to destabilize the shale.

The more generally recognized chemical osmotic force is determined by the relative water activities (vapor pressures) of the drilling fluid and shale pore fluid at downhole conditions. The fluid flow is from the dilute to the more concentrated solution. Therefore, chemical osmosis is opposed by diffusion osmosis when using a water-based drilling fluid. Even if the chemical osmotic force predominates and is extracting water from a shale, diffusion osmosis can cause solutes from water-base drilling fluid to invade the shale and create instability.

Water and solutes can be transferred from a water-based drilling fluid to a shale by diffusion osmosis even when there is no hydraulic pressure differential (borehole and initial shale pore pressure are equal), and there is no chemical osmotic force (activities of the drilling fluid and shale are equal at downhole conditions). In certain situations, the addition of a salt to a water-base drilling fluid to reduce the water activity can cause an increase in the diffusion osmotic force that fully offsets the desired increase in chemical osmotic force. As a result, the inclusion of the salt would not improve shale stability and it might have an undesired effect of restricting the on-site disposal of drilling fluid and drill cuttings.

Destabilizing ionic reactions within a shale can be minimized if a suitable nonionic polyol is used to reduce the activity of a fresh-water drilling fluid so that water is extracted from a shale by chemical osmosis. When using such a fluid to drill a loosely-compacted shale, chemical osmotic effectiveness can be improved by emulsification of a low concentration of a suitable non-aqueous phase. For example, a fresh-water drilling fluid containing methyl glucoside for activity control and emulsified pentaerythritol oleate for permeability reduction prevented hydration and maintained stability of Pleistocene shale from the Gulf of Mexico. Drill cuttings from such a drilling fluid should be environmentally acceptable for discharge at either offshore or land locations.

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Appendix A - DSC Test Procedures

The DSC equipment and test procedures have been discussed in detail in previous publications. 4.6.7 The Gulf of Mexico Pleistocene shale specimens for the DSC tests presented in this paper were cut to a length of 5 inches and drilled with a 1.0-inch diameter, two-bladed tungsten carbide drag bit. The circulation rate while drilling was 1.5 gal/min. After drilling, the bit was retracted and a 0.75-inch diameter tube was inserted into the borehole to simulate drill pipe. The drilling fluid circulation rate was reduced to 1.0 gal/min to provide an annular shear rate of 537.6 1/sec, based on gauge hole.

Preliminary DSC tests of the Pleistocene shale showed it to be extremely sensitive to solids-free water-based fluids. For this reason, the sandpack at the outer periphery of the shale specimen was filled with LVT mineral oil. The pressure in the sandpack was adjusted to the desired initial pore pressure as the shale specimen was subjected to the downhole stresses and temperature. The specimen was then allowed to equilibrate for 24 hours. During drilling fluid circulation, after the specimen was drilled, oil was drained or pumped into the sandpack to maintain constant pressure. This provided a quantitative measure of fluid transport from drilling fluid to the shale. The measurement of fluid transfer from the shale to the drilling fluid was only qualitative, because passage of oil from the sandpack to the shale was hindered by capillary forces.

Appendix B - Drilling Fluid Compositions

Oil-Based Emulsion: $a_{df} = 0.89$	
LVT Mineral Oil	0.8 bbl
16% w/w CaCl ₂ Brine	0.2 bbl
Fatty Acid Emulsifier	4.0 lb
Polyamide Emulsifier	2.0 lb
Lime	3.0 lb
Pierre/Shale (ground)	10.0 lb

Oil-Based Emulsion: $a_{df} = 0.93$ Same as Above Except 12% w/w CaCl₂ Brine

Simulated Pore Fluid: $a_{df} = 0.95$	
Water	0.97 bbl
CaCl ₂	12.0 lb
NaCl	11.0 lb
$MgCl_2$	5.2 lb
KCL	2.0 lb
$\frac{CaCl_2}{10.3\text{-lb/gal CaCl}_2} \frac{Brine: \ a_{df} = 0.72}{Brine}$	1.0 bbl
$\frac{\text{CaCl}_2}{11.2\text{-lb/gal. CaCl}_2} \frac{\text{Brine: } a_{\text{df}} = 0.54}{\text{11.2-lb/gal. CaCl}_2} \text{ Brine}$	1.0 bbl

70% Methyl Glucoside $a_{df} = 0.78$

MEG 207

KOH for pH 9.5 1.0 bbl

70% Methyl Glucoside/Ester Lubricant/Lignite: $a_{df} = 0.80$

MEG 207	0.9 bbl
Finagreen EBL Ester Lubricant	0.1 bbl
Lignite	10.0 lb
KOH	2.0 lb

<u>60% Methyl Glucoside/Ester Lubricant/Lignite:</u> $a_{df} = 0.88$

Same as Above Except MEG 207 Diluted with Water to 60% w/w

<u>60% Methyl Glucoside/Ester Lubricant/NaCl/Starch:</u> $a_{df} = 0.80$

MEG 207	0.77 bbl
10-lb/gal NaCl Brine	0.13 bbl
Finagreen EBL Ester Lubricant	0.10 bbl
Starch	5.0 lb

KOH for pH 10

SI Metric Conversion Factors

bb	1 x	1.58 9873	E-01	=	m^3
${}^{\mathrm{o}}\mathrm{F}$		(°F-32)1.8		=	$^{\mathrm{o}}\mathrm{C}$
ft	X	3.048*	E-01		
Gal/min	X	6.309 020	E-05	=	m ³ /sec
Gram	X	1.0*	E+03	=	kg
in	X	2.54*	E+00	=	cm
Micron	X	1*	E-06	=	m
Lb/gal	X	1.198 264	E+02	=	kg/m ³
psi	X	6.894 757	E+02	=	kPa

^{*}Conversion factor is exact.

TABLE 1 – PROPERTIES OF SHALES TESTED IN GRI STUDIES (from Texaco EPTD)

			MERCURY INJECTION POROSIMETRY		
SHALE	GRAIN DENSITY g/cm ³	BOYLE'S LAW POROSITY %	POROSITY %	CALCULATED AIR Permeability mD	
Gulf of Mexico Pleistocene, West Delta	2.645	27.8	23.9	0.026	
North Sea Cretaceous, Speeton	2.632	18.5	10.8	0.00034	

TABLE 2 - RESULTS OF DSC TESTS

Drilling Fluid	a _{df}	Differential Pressure, psi	Time, hr	Fluid Flux Into Shale ml/hr	Shale Properties Near Borehole Surface %Moisture Hardness	
Oil-Based						
Emulsion	0.89	0	48	-0.06	13	35
Oil-Based						
Emulsion	0.93	0	48	-0.05	13	35
Oil-Based						
Emulsion	0.97	0	48	0.04	12	35
Simulated Pore						
Fluid	0.95	0	7	0.78	21	5
CaCl ₂ Brine	0.70		4.0	0.00	4-5	
	0.72	0	12	0.23	15	0
CaCl ₂ Brine	0.54	0	53	-0.02	13	0
70% Methyl						-
Glucoside	0.78	0	54	-0.02	11	30
70% Methyl						
Glucoside	0.78	200	52	0.09	12	35
70% Methyl Glucoside/Ester Lubricant/Lignite	0.80	200	53	-0.03	12	15
60% Methyl Glucoside/ Ester Lubricant/NaCl/ Starch	0.80	200	52	-0.01	10	40
60% Methyl Glucoside/Ester Lubricant/Lignite	0.88	200	52	-0.01	11	35

Notes: Typical unexposed shale cores had moisture of 11 to 13% and penetrometer hardness of 90-95. Negative fluid flux rates are not quantitative because passage of oil from the sandpack to the shale is hindered.

TABLE 3 – EFFECTS OF DRILLING FLUIDS ON EXCHANGEABLE BASES OF GULF OF MEXICO SHALE

(Hydraulic Pressure Differential = 0)

	Exchangeable Bases, meq/100g			
	<u>Na</u>	<u>Ca</u>	\underline{Mg}	<u>K</u>
Oil-Based Emulsion ($a_{df} = 0.89$)				
Initial Shale	11.0	9.6	5.3	1.1
1/4" from borehole after 48 hours	9.8	8.2	4.1	0.9
Simulated Pore Fluid ($a_{df} = 0.95$)				
Initial Shale	10.7	7.3	4.5	1.0
1/4" from borehole after 7 hours	5.1	12.3	8.2	1.7
$CaCl_2$ Brine ($a_{df} = 0.54$)				
Initial Shale	10.9	7.4	4.5	0.9
1/4" from borehole after 53 hours	0.3	19.5	0.5	0.5
70% Methyl Glucoside ($a_{df} = 0.78$)				
Initial Shale	9.8	7.8	4.2	0.9
1/4" from borehole after 54 hours	5.4	7.9	3.1	6.6

TABLE 4 - ION ANALYSES OF GULF OF MEXICO SHALE PORE FLUID AND SIMULATED PORE FLUID USED AS DRILLING FLUID

(Shale Moisture Content: 14.9g/100g of Dry Shale)

		Extractable			Ions in Simulated
	Bound Ions,	Ions,	Total Ions,	Total Ions,	Pore Fluid Used
	meq/100g	meq/100g	meq/14.9 ml	meq/L	as Drilling Fluid,
	of Dry Shale	of Dry Shale	of pore water	of pore water	meq/L
Cations					
Calcium	7.3	Tr	7.3	490	640
Sodium	10.7	3.8	14.5	973	560
Magnesium	4.5	0.8	5.3	356	320
Potassium	1.0	Tr	1.0	67	80
Other		0.5	0.5	34	
Total Soluble	23.5	5.1	28.6	1,920	1,600
Anions					
Chloride		2.2	2.2	169	1,600
Sulfate		1.8	1.8	146	
Carbonate		1.1	1.1	85	
Clay	23.5				
Total Soluble		5.1	5.1	400	1,600

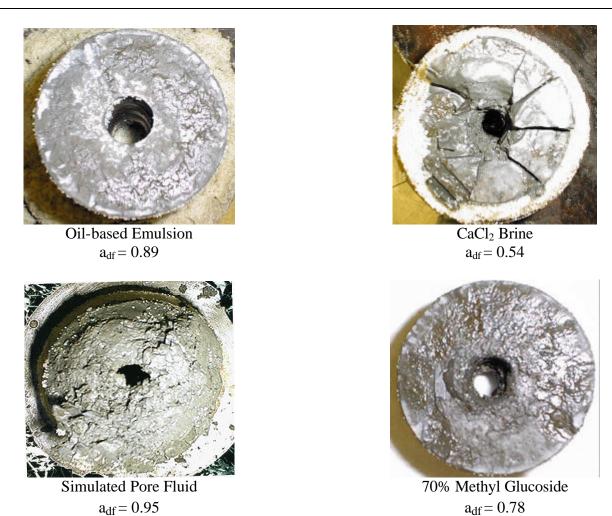
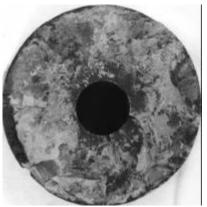


Fig. 1 – Gulf of Mexico shale specimens after exposure to various drilling fluids with zero hydraulic pressure differential.



70% Methyl Glucoside $a_{df} = 0.78$



70% Methyl Glucoside/ Ester Lubricant/ Lignite $a_{df} = 0.80$

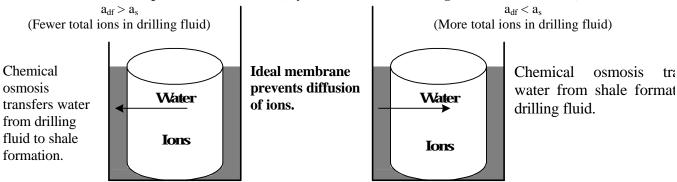
Fig. 2 – Gulf of Mexico shale specimens after exposure to various drilling fluids with hydraulic pressure differential of 200 psi.

No Membrane (Water-Based Drilling Fluid/Sand Interface)

 $a_{\rm df} > a_{\rm s}$ (Fewer total ions in drilling fluid) (More total ions in drilling fluid) Hydrated ions of Water diffuses Water diffuses each species from drilling from sand formation diffuse from fluid to sand high to low to drilling fluid. Water Water formation. concentration. Ions Tons

There is no change in sand formation pore pressure.

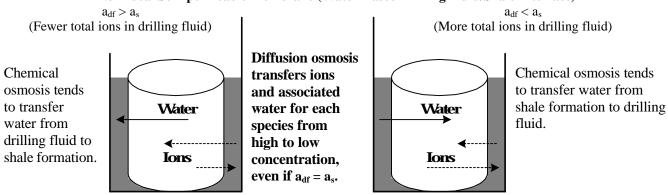
Ideal Semipermeable Membrane (Hydrocarbon-Based Drilling Fluid/Shale Interface)



Shale pore pressure near borehole surface is increased, with dissipation to the far field.

Shale pore pressure near borehole surface is decreased, with shale water content replenished from the far field.

Non-Ideal Semipermeable Membrane (Water-Based Drilling Fluid/Shale Interface)



The change in shale pore pressure near the borehole surface and the net transfer of water depend upon: (1) the water activities of the drilling fluid and shale pore fluid; (2) the concentrations of the ions in both the drilling fluid and shale; and (3) the shale/fluid ion selectivities at downhole conditions.

Fig. 3 - Chemical and diffusion osmosis: transfer of water and inorganic ions between drilling fluid and formation when the drilling fluid pressure is equal to the far-field formation pore pressure.

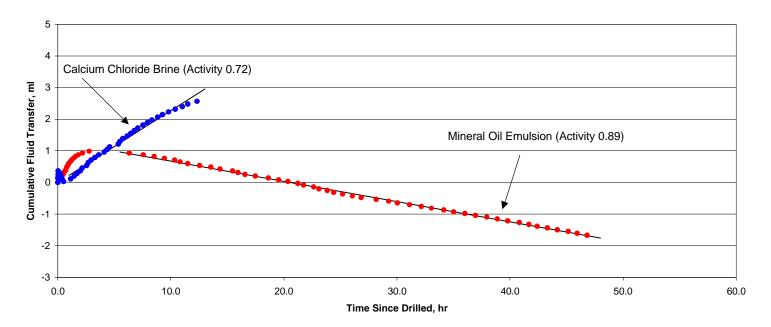


Fig. 4 – Fluid transferred at the sandpack at the outer periphery of Gulf of Mexico shale specimen when exposed to drilling fluids with no hydraulic pressure differential.