Asphaltic Shale Coating Agents

by

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Asphaltic Shale Coating Agents

Approved by
Supervising Committee:

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Dedication

I would like to dedicate my work to my parents who helped me through every stage. I am very grateful to be their son.
Acknowledgements

I would like to thank my supervisor Dr. M. E. Chenevert for his support, guidance and financial support towards my Master’s Degree. I highly appreciate the time, advice and guidance he provided me in the last year. I thank Dr. Mukul Sharma, my co-supervisor for his research inputs and help with this thesis.

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Abstract

Asphaltic Shale Coating Agents

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The University of Texas at Austin, 2004

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Environmental regulations concerning the use of oil-based fluids have motivated the development of water-based muds that contain asphaltic shale coating agents. In particular, drilling guidelines discourage oil-based fluids due to their negative environmental effects. Water-based muds offer a much cheaper and environmentally acceptable solution.

The problem with the use of water-based muds is the induced wellbore instability due to the tendency of shales to interact with water. The shales consequently swell or shrink and may eventually cause wellbore collapse. A variety of additives are currently available that claim to be successful in stabilizing shales. Two such agents that are asphaltic in nature, namely Bore-plate and Soltex, have been evaluated in this study for their role in shale stabilization.

This thesis is an effort to evaluate the claim that Bore-plate and Soltex reduce the permeability of shale, by physically coating the surface of the shale. An experimental laboratory-scale apparatus was used. The results to date indicate that the ability of the
above two asphaltic coating agents to reduce the permeability of shale is dependent on temperature. No reduction in shale permeability was observed at room temperature. At elevated temperatures (240°F) the additives reduced the permeability of the shale by 16.78% for Pierre shale. No reduction in permeability could be achieved for Arco China shale. The results indicate that these additives have very limited potential for reducing water & ion influx into shales.
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Chapter 1: Introduction

Drilling through shale formations has traditionally been difficult. The tendency of shales to swell or shrink and consequently cause borehole collapse often leads to expensive drilling delays and thereby increases the costs involved in drilling and completing wells. The common remediation measure is to use a balanced-activity oil-based mud or synthetic-based mud. The last ten years have witnessed increased environmental concerns with the use of such muds and the disposal of cuttings. This along with increased expenses involved in using oil-based muds are the motivation behind evaluating water-based muds and the possibility whether shales can be drilled safely with water-based muds. If we compare the advantages of water-based muds to their oil-based counterparts, the incentive is that they can be very easily mixed at the site and are very easy to dispose of. This is besides the fact that they only cost a fraction of oil-based muds.

Shale is defined as a fine-grained sedimentary rock having a laminated and often friable structure. Unstable shales have always plagued the industry (Chenevert, 1969). All boreholes drilled into shale formations experience some degree of washout, which may be tolerable in some wells but completely uncontrollable in some. The reasons for instability may be numerous ranging from mechanical, chemical to physical reasons. Mechanical problems include borehole erosion by high annular velocities, adverse hydraulic stresses caused by high annular pressures, hole collapse from high surge and swab pressures because of excessive filter cake, and accelerated erosion resulting from drill-string movement. Chemical alteration problems include hydration, dispersion and disintegration of shales due to interaction with the water-phase. Physical instability problems include the spalling and rock burst of shales caused by subnormal pressure or
overpressure relationships of hydrostatic and formation pressures. Fracture and slippage along bedding planes of hard, brittle shales and the collapse of fractures shales above deviated holes are also physical problems encountered during drilling of troublesome shales. This problem also occurs in non-deviated holes during drilling of over-pressured shales. Wellbore instability problems are often referred to as sloughing, heaving, spalling, mud balls, mud rings etc.

The answer to the question, “why shales behave in such a manner?” may be explained by considering shale-fluid interaction, i.e., the exchange of water molecules and solutes/ions. The main driving forces for this interaction are hydraulic pressure gradients and chemical potential gradients that transport water molecules and ions between drilling fluid and the shale. This changes the magnitude of hydration stresses and increases tensile stresses. Pore-pressure elevation will reduce the near-wellbore effective stresses that hold the material together. Near-wellbore water content and cementation integrity changes the formation strength. Clearly, control over near-wellbore stresses and formation strength is essential to maintain stability.

The remedies to problems created by shales (with water-based muds) are limited. The commonly used procedure for fractured shale is to use effective sealing agents, lower mud weight and thixotropic drilling fluids. An inhibitive mud may be used when shales are encountered. Reduction of drilling fluid water activity is the heart of these inhibitive muds. This reduction is brought about by adding electrolytes: seawater bentonite muds, saturated salt-polymer (xanthan, guar), KCL or NaCl-polymer (PHPA), fresh water calcium treated muds (lime, gypsum).

A new type of drilling fluid based on a substituted sugar, methyl glucocide, is currently being looked into because of its ability to form low activity muds with considerable reduction in water/ion movement. This product is called “ULTRADRILL”
and is marketed by MI-Swaco. “PERFORMAX” is Baker Hughes Drilling Fluids' new 3rd generation high-performance water-based drilling fluid that is designed to emulate performance characteristics previously achieved only with emulsion based drilling fluids. These attributes include shale inhibition (reduced pore pressure transmission), suppressing clay swelling and hydrations. “HYDRO_GUARD” is a Baroid product claimed to be a good inhibitive WBM system. Nevertheless, it is difficult to balance water activity of shale with that of the mud through the entire drilled depth of the well because the shale water activity is not known and it varies with depth and mineralogy. Besides, when the drilling fluid activity is lower than that of shales it might dry out the pore fluid from within the shale, which may cause it to crumble depending on its type.

The other option available for shale stability is reducing the effective permeability of the shale. It seemed that soluble additives that would from a permeability barrier at the shale surface or within micro-fractures and prevent any water/ion transport in to shale, would be a good prospect. Such additives ‘Silicate’ and ‘Alplex’ were developed. The working mechanism of silicate and Alplex muds is permeability reduction attained by reaction of specific chemicals with the shale lithology, making it practically impermeable. The disadvantages of the above systems are: the literature available on such muds is relatively scarce; the reservoir characteristics after their use are unknown, and the possibility of the mud severely affecting other formations. Cationic polymers, which are strongly adsorbing, can also reduce permeability. But a suitable polymer has not been found.

This leaves us with the focus of this study; Shale formation might be completely isolated by creating an impermeable seal, using asphaltic derivates like ‘Gilsonite’ and ‘Soltex.’ This thesis is basically the evaluation of these two additives as a potential shale stabilizer by permeability reduction. There is very limited data available about the
performance of these additives. A few tests have been performed using the ‘Down-Hole Simulation Cell’ at the OGS testing facility. The expense involved limits the extent of tests that can be performed. There is a need for creating a laboratory-scale apparatus that can test additives under conditions of temperature and pressure. This thesis is a continuation (Duantem, 2003) and incorporates a previously developed experimental setup with a newer “hybrid” filtration cell.

During early on testing the inability to reproduce data and severe buckling of shale samples were the problems faced. The remedy of these problems led to the fabrication of a new filtration cell that would allow for testing muds. The same cell can be used in the future for testing other muds, salt solutions and even OBM. The new cell provided a much better degree of success with the two different sets of shale samples that were used in the experiments.
Chapter 2: Literature Review

Wellbore instability in shales is the prime technical problem in oil and gas drilling, with lost-time and trouble costs for the drilling industry conservatively estimated at $500 million/year (Van Oort et al, 1996). Shales make up over 75% of the drilled formations and over 70% of borehole problems are related to shale instability (Lal, 1999). Moreover, the industry is facing new technical and environmental challenges associated with drilling increasingly difficult wells (e.g. Horizontal multilateral and extended reach wells) and replacement of poorly biodegradable oil based muds (OBM’s) that are technically superior but environmentally unacceptable. Water based muds (WBM’s) offer a cheaper environmentally acceptable alternative, but have not kept up with the advances in the drilling industry in the performance criteria’s such as rate of penetration, thermal stability and lubricity. Lack of understanding about shale/drilling fluid interaction can lead to costly trial and error multi-well strategy and to what is often called an expensive learning curve before arriving at a satisfying solution.

Many authors have discussed the background of shale and drilling fluid interactions. The following are the interaction mechanisms involved (Chenevert, 1969; Ewy et al, 2000): mechanical stresses, fractured shales, capillary pressure, osmosis, hydraulic (advection), swelling/hydration pressure, pressure diffusion and fluid penetration. A concise explanation is that adsorption produces a net flow of water that causes the clay layers to separate, thus inducing internal expansive stresses. These reduce the shale strength and cause it to collapse.

Gauge boreholes can be drilled through both hard and soft shale using balanced-activity oil-continuous muds (Chenevert, 1970). Using OBM has always saved considerable drilling time and hazardous conditions such as stuck pipe and high torque
were eliminated. According to Mody et al, (1993) the two predominant factors why OBM’s are better in stabilizing shales are (1) a continuous oil phase exerts a confining pressure on the borehole wall since the mud column pressure typically exceeds the formation pressure and does not exceed the threshold capillary entry pressure, and (2) chemical potential between the OBM and the shales result in the selective transport of water in and out of the shale. Water adsorption can be avoided by balancing the activity of the internal aqueous phase of the OBM and the in-situ water activity of the shale (Chenevert, 1970). As described before environmental concerns and expenses involved are the reasons to evaluate WBM.

In order to have a better understanding of WBM we need to understand the internal mechanisms and driving forces involved in shale and drilling fluid interactions. The filtrate flow \( V_f \) into shales, which is described in Eqn (2.1) as below (Van Oort et al, 1994; A):

\[
V_f = \frac{k \left( \Delta P - \sigma \Delta \Pi \right)}{\eta} \tag{2.1}
\]

Where \( k \) is the shale permeability, \( \Delta P \) is the hydraulic overbalance, \( \sigma \) is the membrane efficiency, and \( \Delta \Pi \) is the osmotic pressure defined by:

\[
\Delta \Pi = \frac{RT}{V_w} \ln \left[ \frac{a_w^{sh}}{a_w^{df}} \right] \tag{2.2}
\]

Where \( T \) is the temperature, \( R \) is the gas constant, \( V_w \) is the partial molar volume of water, and \( a_w^{sh} \) and \( a_w^{df} \) are the shale and drilling fluid water activities respectively. We can conclude that there are four mechanisms that can reduce \( V_f \):

1. Reducing the flow of mud filtrate by increasing the filtrate viscosity \( \eta \)
2. Reducing the flow of mud filtrate by reducing the shale permeability $K$;

3. Balancing the flow of mud filtrate into the shale driven by overbalance $\Delta P$ by an
   backflow of pore water driven by the effective osmotic pressure $\sigma \Delta \Pi$

4. Combining the above mechanisms.

Soluties that can increase the fluid viscosity significantly and also pass through the
narrow shale pores have not been found (Van Oort et al, 1994; B). This implies almost all
attempts are either at reducing the shale permeability or balancing the overburden and
osmotic pressures. A review of recent advances is presented.

A commonly used inhibited WBM is a KCl mud that contains xanthan gum,
partially hydrolyzed polyacrylamide (PHPA), polyanionic cellulose (PAC), and possibly
a polyglycol. This combination has provided varying success in limiting the rate of
hydration, pore-pressure build-up and shale weakening, enough to allow a shale section
to be drilled and cased, but with very little success in high-angle and extended-reach
wells (Simpson et al, 1994). Inorganic salts can reduce mud activity, but the hydrated
ions are small enough to penetrate shale pores. With the shale presenting a very
inefficient semi permeable membrane, little osmotic pressure can be developed (Mody et
al, 2002). Reducing the activity of brine muds can reduce the water flux into shale, but
mud activities far lower than presently used would be required to prevent such influx in
most instances (Schlemmer et al, 2002). Attempts were made with water-soluble straight-
chain polyalcohols called glycerol muds. These did not provide any degree of
stabilization (Simpson et al, 1994).
A new WBM claimed at having an excellent chance of replacing OBM’s is methylglucoside (MEG) (Chenevert et al, 1998). MEG is a derivative of glucose, supplied as liquid containing 70% solids. It is manufactured from corn starch and is classified “readily biodegradable.” MEG systems claim lubricity coefficients, filtration rates and storage ability similar to OBM’s, with the added advantage of cuttings being environmentally acceptable. This water-soluble organic monomer having multiple hydroxyl groups on a cyclic structure capable of reducing water activity of mud and may be the only alternative to OBM’s currently available.

Ammonia-derived chemicals including amines, amides, polyamides, imines, nitriles and amino acids are used in many water based drilling fluid components (Schlemmer et al, 2003). Ammonium chloride was tested more than 30 years ago as a shale inhibitor. Diammonium phosphate has been used for more than 20 years. Organoaamines were recently introduced. Relatively simple organic amines were first added to drilling fluids 15 years ago to enhance cuttings and wellbore stability. Offering performance approaching that of a potassium cation, simple amines effectively carry on in many regions for specific applications.

Drilling muds based on soluble silicates for the control of shale that sloughs into the wellbore in excessive quantities, were introduced in the 1930’s. These muds based on high concentrations of soluble silicates were successful in drilling over 100 wells in the gulf coast area, but were difficult to control because of their high rheologies. Limequebracho muds replaced them after a decade. Silicate-based drilling fluids are claimed to stabilize both intact and micro-fractured shale and chalk formations (Van Oort et al, 1996; B). Silicates protect shales by in-situ gelling and precipitating when exposed to neutral shale pore fluid and/or polyvalent ions involving both permeability reduction and balancing overburden pressure by osmotic pressure mechanisms. The activity of the fluid
phase should at least match the activity of the shale. Silicate muds are environmentally acceptable, inexpensive and can be formulated to withstand contamination. Reason they are not being extensively used would be that their reservoir damage characteristics have not been evaluated. Alplex is a product that works on very similar lines as silicate muds forming insoluble precipitates blocking shale pores. When alplex-treated fluids enter the shale pore network, aluminum hydroxides are precipitated that transform into crystalline forms becoming part of the shale crystal structure, thereby blocking pore pressure build-up in shales (Alplex, 1996).

The focus of this thesis is asphaltic derivatives that claim to create impermeable hydrophobic seals. At a specific temperature and pressure, asphalt will extrude and plug the shale pores and micro-fractures to minimize filtrate invasion, and bond the matrix particles together to prevent sloughing (Davis et al, 1998). If the above concept holds true there would be no need to balance the activity of the drilling fluid. Bore-plate is a blended Gilsonite, which was developed by the American Gilsonite Company. Gilsonite is a naturally occurring solid hydrocarbon material, which is classified as an Asphaltite (Davis et al, 1998, 2002; The American Gilsonite Company 1991). It is a relatively pure natural product occurring in a mineral called Uintaite. It has a low specific gravity with glossy-black appearance, and softening points ranging from 300 to 450 °F. The chemical and physical properties of Gilsonite vary and depend strongly on the Uintaite source. Gilsonite and other asphaltic-type products have been used worldwide in water-based drilling fluids as a borehole stabilizer. However, Gilsonite was reported by Cagle et al (1972) to be superior to other asphalts because of its low solubility, fewer impurities, and higher softening point. Cagle et al (1972) reported Gilsonite to be superior asphalt due to its low solubility, high softening point and inherent purity. “It is composed of high and low softening point Gilsonite, causticized lignite, and a strongly lipophilic non-ionic
surfactant. Because Gilsonite is not water-wettable, a strong surfactant is required to provide good wettability and rewettability, making Gilsonite remain dispersible in the water-based drilling fluid system. The causticized lignite improves borehole stabilization at high temperatures, and also the dispersability of Gilsonite” (Duantem, 2003). They found that this combination provides an effective water-wettable additive that is easily dispersible, remains water-wet, and can improve wellbore stability in a broad range of borehole temperatures from ambient up to 450 °F (The American Gilsonite Company, 1991). Bore-plate used in our project is blended Gilsonite, a mixture of high and low softening point Gilsonite at a ratio of 1:2 by weight.

Soltex is a product of the “Drilling Specialties Company.” It is made from sulphonated asphalt. This sulfonating makes it partially soluble in water and become an anionic compound. According to the manufacturer, Soltex is able to attach itself to the charged edges of the shales, thereby reducing water penetration. “Also, its wide range of particle sizes is claimed to considerably contribute to shale stability improvement since these particles can fit and effectively plug off a wide range of pore sizes in a shale matrix” (Duantem, 2002). All of these characteristics, including surface charge, a wide range of particle sizes, and its partial solubility, are claimed to make Soltex more effective than other asphalts in shale stabilization. Davis et al (1998) suggests that sulfonated asphalt penetrates more deeply into fractures because of its higher solubility, and does not plate the borehole as effectively as insoluble products such as Gilsonite. The results obtained in this thesis will provide a comparison of the above additives.

Because Gilsonite and the asphaltic-type additives require temperature and pressure for its effectiveness in shale stabilization, it has been difficult to evaluate these products by laboratory tests that were normally conducted under ambient conditions (Davis et al, 1998).
Chapter 3: Test Equipment and Experiment Design

3.1 Experimental Apparatus and System Diagram

3.1.1 High-Pressure, High-Temperature (HPHT) Static Filtration Cell

Figure 3.1 shows the schematic of the new HPHT static filtration cell used in this study. This cell has three basic parts: a cover, a body and a base. The body is designed to be diametrically hollow to accommodate a desired amount of drilling mud. The base holds the shale sample and facilitates filtration. The cover encloses the body and provides for the upstream pressure to be applied on the mud enclosed in the body.

A unique feature of the filtration cell is the inclusion of a ‘bleed plate’ between the mud and the shale sample. This bleed plate contains many 1/16” holes and essentially is the spine for the shale sample and prevents the sample from cracking under pressure and at the same time allows the mud to remain in contact with the shale sample. The filtrate coming out of the shale sample is channeled through a wire mesh and out of the base of the filtration cell. While conducting the filtration test, the top fluid pressure is applied by a nitrogen gas cylinder that supplies pressurized gas through the inlet on the top of the cell.

During the development of the filtration cell, three design modifications were incorporated (post-design stage) in to the base portion. The first stage of the modification involved the circular groove below the shale sample that held the porous plate, intended to channel filtrate out of the cell. Inconsistency in the groove caused a ‘digging effect’ in to the soft shale, which eventually cracked the shale when the cell was assembled. The second modification involved decreasing the height of the lip of the groove; this meant that the thickness of the porous plate was greater than the lip. This did not prevent the
shale from cracking, from reasons that could not be explained. This prompted a third modification that completely discarded the groove and porous plate arrangement. Two circular wire mesh screens of the same diameter as the shale were placed under the shale sample, which channeled the flow to the outlet stem of the cell.

Figure 3.1: High-pressure, high-temperature static filtration Cell
3.1.2 Flow Diagram of Testing System

Figure 3.3 shows the complete flow diagram of the testing system, which has essentially remained the same as Duantem’s system (Duantem, 2003). This system consists of the filtration cell, the gas cylinder, the hand pump, the vacuum pump, the pressure transducers, and valves. Except for the filtration cell all other components are pressure management devices. Fig.3.2 shows the real life setup of laboratory equipment. As shown in figure 3.3, a pore-fluid reservoir is located underneath the shale disk sample through the connection of the bottom outlet stem. This reservoir is filled with a simulated pore fluid before the flow test begins. Pore pressure is increased as filtration is in process. A condition of no increase in pore pressure indicates zero fluid movement into the shale sample and zero permeability.

Figure 3.2: Apparatus for data monitoring
Figure 3.3: Testing System Flow Diagram (from Duantem, 2003)
3.2 Testing System Components

1. **Filtration Cell** – This is the main experimental apparatus for this filtration study.

2. **Vacuum Pump** – Air in the bottom pore fluid reservoir is evacuated using a Welch Duo-Seal Model 1402 vacuum pump so as to provide a liquid only condition. After air evacuation, the pore fluid from the pore fluid source, as shown in Figure 3.3, fills up the bottom reservoir by atmospheric pressure.

3. **Nitrogen** – The pressure source for the test fluid on the top of the shale sample is a standard compressed nitrogen cylinder. (Distributed by Matheson Tri-gas, Irving Texas).

4. **Hand Pump** – This is a Temco positive displacement, hand operated pump Model 25-10-HAT that supplies the pressure source for the pore fluid reservoir at the bottom of the shale sample. This is the most essential component to monitor the filtration rate out of the shale and maintain a constant pressure differential across the shale. The pump reads filtration rate directly in ml with a least count of 0.0005 ml.

5. **Valves (V₁-V₈)**
   - **V₁** – Top Pressure Bleeding Valve
   - **V₂** – Flow-Controlled Valve of the electrical pump
   - **V₃** – Top-Bottom Isolating Valve, this valve provides the flexibility of a pressure source supplier. The top and the bottom of the shale disk can directly communicate by opening this valve as shown in Figure 3.3.
   - **V₄** – Hand Pump Isolating Valve
   - **V₅** - If the test were to be designed to monitor change in pore pressure instead of keeping it constant during the filtration, the valve V₅ is closed in order to minimize the
volume of the pore fluid reservoir to reduce testing time (time for pore pressure to reach equilibrium).

\( V_6 \) – Bottom Pressure Bleeding Valve, this valve is kept in closed position at all times during the filtration test.

\( V_7 \) – Vacuum Pump Bleeding Valve (3-way valve)

**6. Pressure Transducers (G\(_1\)-G\(_3\))**

- G\(_1\) – Top Fluid Pressure Measurement
- G\(_2\) – Pore Pressure Measurement for leak testing purposes
- G\(_3\) – Pore Pressure Measurement on bottom of sample

### 3.3 Equipment Pressure Testing

Equipment integrity is extremely imperative to precise results, especially in the downstream side of the setup. The downstream side is extremely pressure-sensitive, the order of 50 psi for 0.001ml of filtrate flux per minute. For this reason a pressure integrity test was performed every month during the period of investigation. Pressure testing consisted of testing all of the tubing connections including pressure transducers and valves, while isolating the filtration cell from the system. The pressure test was performed at 1000 psig at room temperature over a 24-hrs period.

Testing the integrity of the filtration cell and the O-ring seals inside the cell to ensure there is no communication between the top and the bottom part of the shale sample is also performed. This test is performed similar to a regular filtration experiment with the shale sample switched by a brass plug core of the same dimensions as that of the shale. A pressure differential of 300 psig (500 psig upstream and 200 psig downstream) is applied for 24 hours. For the above tests a loss factor of less than or equal to 1 psig per hour is acceptable is considered acceptable.
3.4 Elevated Temperature Testing

The test matrix required six experiments to be done at 240°F. For this reason a single wall Transit Oven manufactured by BLUE M, Illinois USA with a rating up to 200 degrees Celsius was used.

3.6 Shale Samples

Two sets of shale samples; Arco China and Pierre were tested. It was ensured that a set of experiments (three in number) used shale samples obtained from the same parent core. The water activity of the Arco China shale was calculated to be around 0.82 and for Pierre it is assumed to be very close to 1. It needs to be noted that from a rock mechanics point of view the above shale samples are very distinct from each other. Pierre shale is from an outcrop and Arco China shale is obtained from a depth of 17,500 feet. The Pierre is comparatively more permeable and less “tight”. X-ray diffraction analysis and adsorption curves for the shales are described below.
TABLE 3.1: Mineralogical composition and water activity of Arco and Pierre I shales

<table>
<thead>
<tr>
<th>X-Ray Diffraction Analysis % by weight</th>
<th>Arco Shale</th>
<th>Pierre Shale</th>
</tr>
</thead>
<tbody>
<tr>
<td>Quartz</td>
<td>23.6</td>
<td>19</td>
</tr>
<tr>
<td>Feldspar</td>
<td>4.0</td>
<td>4.0</td>
</tr>
<tr>
<td>Calcite</td>
<td>-----</td>
<td>3</td>
</tr>
<tr>
<td>Dolomite</td>
<td>1.2</td>
<td>7</td>
</tr>
<tr>
<td>Pyrite</td>
<td>2.4</td>
<td>2</td>
</tr>
<tr>
<td>Siderite</td>
<td>4.1</td>
<td>1</td>
</tr>
<tr>
<td>Chlorite</td>
<td>3.6</td>
<td>4</td>
</tr>
<tr>
<td>Kaolinite</td>
<td>5.7</td>
<td>11</td>
</tr>
<tr>
<td>Illite</td>
<td>15.0</td>
<td>19.0</td>
</tr>
<tr>
<td>Smectite</td>
<td>11.0</td>
<td>17</td>
</tr>
<tr>
<td>Mixed layer</td>
<td>29.4</td>
<td>49</td>
</tr>
<tr>
<td>Total</td>
<td>64.7</td>
<td>64</td>
</tr>
<tr>
<td>Water Activity</td>
<td>0.82</td>
<td>0.98</td>
</tr>
</tbody>
</table>

Figure 3.4: Adsorption Isotherms
3.6 **Shale Sample Preparation**

Preparation of shale samples has essentially remained standard over time and is described below. The cross-sectional area of the shale sample is square (1.55” X 1.55”) with 0.25-in thickness. An exception to the square cross-section would be for the first three experiments of the test matrix, wherein a circular shale core of 2.25” diameter was used. The epoxy surrounds the shale to form a circular disc of diameter 2.5”, which is required for the filtration cell to provide a good sealing. This shale disk was prepared by having a plastic ring sealed with epoxy around the external surface to minimize air exposure. The plastic ring, which is made from heat-resistant material, i.e. Polycarbonate, adheres to the shale sample by epoxy. The detailed sample preparation method follows.

The shale core sample was first cut in to a square core (or circular). This core was now ready for sample epoxy sealing.

Prepare epoxy by mixing epoxy resin and hardener at the ratio 1:1 by weight. Mix it thoroughly.

Pour the epoxy inside a 2.5 x 2.25-in plastic tubing that has a bottom plate.

Place the shale core inside the tubing and centralize the core with 4 1/16-in plastic rods.

Leave epoxy to cure for 24 hrs. Fig 3.5 shows a picture of a shale core.

Slice this shale core to a 0.25-in disk with a circular saw.

After following the above procedure, the shale disk sample was obtained. To prepare these shale samples for testing, they were first placed in Escaid oil containers (completely immersed) to prevent any damage.
Figure 3.5: Shale core in epoxy
Chapter 4: Test Design and Procedure

4.1 Test Objectives

The overall test design is to run a static filtration test on given shale with a constant pressure differential of 300 psig. The constant pressure differential is essential for the applicability of Darcy’s equation for flow through a permeable medium. It involves two different test fluids classified as top and bottom test fluids. The top test fluid is a water-based mud with the necessary shale-coating agent in solution depending upon the test requirement. The bottom test fluid is basically brine at the same water activity as that of the shale. These are described in detail below.

4.2 Test Fluids

4.2.1 Top Test Fluid

In order to compare the results from these experiments with the previous studies, the same mud formulation was used. It was a freshwater lignosulfonate mud having a composition as shown in Table 4.1. The concentration of Bore-Plate and Soltex added to the mud is thrice that recommended by the manufacturers. Tripling of concentration was performed to increase the probability of the additive functioning the desired purpose. For the referenced mud, neither Bore-Plate nor Soltex was added in the formulation. The filtrate is essentially the brine in the water-based mud and corresponding viscosity values were used in the calculation of permeability values of the shale.
TABLE 4.1: Composition of water-based mud

<table>
<thead>
<tr>
<th>Component</th>
<th>Quantity</th>
<th>Unit</th>
</tr>
</thead>
<tbody>
<tr>
<td>Deionized water</td>
<td>0.87</td>
<td>bbl</td>
</tr>
<tr>
<td>Bentonite</td>
<td>18.0</td>
<td>lbm/bbl</td>
</tr>
<tr>
<td>KOH</td>
<td>(Variable for pH adjustment to 9.5)</td>
<td></td>
</tr>
<tr>
<td>Lignosulfonate</td>
<td>2.6</td>
<td>lbm/bbl</td>
</tr>
<tr>
<td>Lignite</td>
<td>5.0</td>
<td>lbm/bbl</td>
</tr>
<tr>
<td>Rev Dust</td>
<td>40.0</td>
<td>lbm/bbl</td>
</tr>
<tr>
<td>Bore-Plate/ Soltex</td>
<td>15.0/18.0</td>
<td>lbm/bbl</td>
</tr>
</tbody>
</table>

TABLE 4.2: Rheology of mud system

<table>
<thead>
<tr>
<th>Mud</th>
<th>$\theta_{600}$</th>
<th>$\theta_{300}$</th>
<th>Plastic Viscosity (cp)</th>
<th>Yield Point (lb/100 ft$^3$)</th>
<th>Gel strength (lb/100 ft$^3$)</th>
<th>10-sec</th>
<th>10-min</th>
<th>API water loss (ml)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reference water-based mud</td>
<td>35</td>
<td>25</td>
<td>10</td>
<td>15</td>
<td>11</td>
<td>15</td>
<td>9.2</td>
<td></td>
</tr>
<tr>
<td>Bore Plate-treated mud</td>
<td>33</td>
<td>21</td>
<td>12</td>
<td>9</td>
<td>9</td>
<td>14</td>
<td>7.3</td>
<td></td>
</tr>
<tr>
<td>Soltex treated mud</td>
<td>34</td>
<td>19</td>
<td>15</td>
<td>4</td>
<td>5</td>
<td>10</td>
<td>7.4</td>
<td></td>
</tr>
</tbody>
</table>

4.2.2 Bottom Pore Fluid

The reservoir underneath the shale sample was completely filled with a simulated pore fluid so that it behaved as the shale fluid reservoir. The simulated bottom pore fluid was basically a salt solution at the same water activity as that of the shale sample. This design minimized the interactions between the shale and the bottom pore
fluid. This allowed the interpretation of pore pressure change during the test as only a result of the interactions between the top test fluid and the shale sample.

Sodium Chloride (NaCl) solution at 0.85 and 0.98 water activity’s were used for the tests with Arco China and Pierre shales respectively.

4.2.3 Fluid Preparation

4.2.3.1 Top Test Fluid Preparation

The referenced water-based mud was prepared with the above formulation in a large batch (3000 ml of freshwater) using the Premier Mill Corp mixing dispersator. Then, a small amount of this mud (350 ml) was taken and mixed using a laboratory mud mixer with Bore-Plate or Soltex to prepare the additive-treated mud for each test. Mud preparation followed this procedure:

Mix deionized water with Bentonite for 30 minutes.

Adjust pH of the mud to +/- 9.5 with KOH solution (20% by weight).

Keep mixing mud for at least 24 hrs (overnight) to allow the Bentonite to fully hydrate before being used.

After overnight shearing, the base mud was mixed with the other components. Add Lignosulfonate, Lignite, and Rev dust. Adjust pH with KOH to +/- 9.5.

Keep shearing the mud for at least six more hrs to allow all the ingredients to thoroughly mix.

Now, the referenced water-based mud is ready for use. To prepare a Bore-Plate/Soltex-treated mud, take 350 ml of this referenced base mud.

Add Bore-Plate or Soltex to this 350-ml mud. Adjust pH with KOH to +/- 9.5.
Keep shearing mud for 2-3 hrs. After shearing, the Bore-Plate/Soltex-treated mud is ready for use.

Perform standard rheology and fluid loss tests.

Prior to using the mud in the experiments, the mud was again sheared with the laboratory mud mixer for 10 minutes immediately before its use. Also, the pH was adjusted with KOH to +/- 9.5.

4.2.3.2 Bottom Pore Fluid Preparation

To prepare the NaCl salt solution at 0.85 water activity, 19% by weight of NaCl crystals and for 0.98 water activity, 4% by weight of NaCl was mixed with deionized water. After the desired salt solution was obtained, it was stored in the corresponding-controlled relative vapor pressure desiccators for a week to ensure that the solution achieved the respective water activity.

4.3 TEST CONDITIONS

The shale static filtration tests were performed with Bore-Plate-treated mud and Soltex-treated mud. Their results were compared with those of the referenced water-based mud at the same conditions, i.e. temperature and pressure to investigate the effects of those two additives. Testing at various conditions allowed us to evaluate the effects of the test conditions on the performance of the products.

4.3.1 Temperature

The product “Bore-Plate” that had been selected for the tests has a low softening point. Temperature required for this additive to work is in the range of 180-250 °F. It is for this reason the test matrix incorporated tests at 240 °F. The second product tested,
“Soltex” is a sulfonated asphalt additive that has been claimed to act as a shale inhibitor without heat requirement.

4.3.2 Pressure

Differential pressure causes filtration to occur and this allows Bore-Plate or Soltex to form a coating on the shale surface and to form plugging of the shale pores. All of the tests were designed at 300-psig differential pressures, which is a typical down-hole overbalance condition in the field. The effect of differential pressure was not investigated in this study.

4.4 Test Matrix

A total of 12 primary tests were successfully completed under the conditions as shown in Table 4.2. Miscellaneous, unsuccessful and practice tests and their results have been presented in a separate chapter. Sets of three experiments per shale sample at a fixed temperature were performed. Two temperatures 70°F and 240°F were considered. The temperature limit of the epoxy is 250°F, therefore further elevated temperatures were not pursued.
<table>
<thead>
<tr>
<th>Test No</th>
<th>Differential Pressure (psi)</th>
<th>Temperature (°F)</th>
<th>Shale</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Fresh WBM</td>
<td>300</td>
<td>70</td>
</tr>
<tr>
<td></td>
<td>0.85-Aw NaCl</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>Fresh WBM/Bore-Plate</td>
<td>300</td>
<td>70</td>
</tr>
<tr>
<td></td>
<td>0.85-Aw NaCl</td>
<td></td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>Fresh WBM/Soltex</td>
<td>300</td>
<td>70</td>
</tr>
<tr>
<td></td>
<td>0.85-Aw NaCl</td>
<td></td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>Fresh WBM</td>
<td>300</td>
<td>240</td>
</tr>
<tr>
<td></td>
<td>0.85-Aw NaCl</td>
<td></td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>Fresh WBM/Bore-Plate</td>
<td>300</td>
<td>240</td>
</tr>
<tr>
<td></td>
<td>0.85-Aw NaCl</td>
<td></td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>Fresh WBM/Soltex</td>
<td>300</td>
<td>240</td>
</tr>
<tr>
<td></td>
<td>0.85-Aw NaCl</td>
<td></td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>Fresh WBM</td>
<td>300</td>
<td>70</td>
</tr>
<tr>
<td></td>
<td>0.98-Aw NaCl</td>
<td></td>
<td></td>
</tr>
<tr>
<td>8</td>
<td>Fresh WBM/Bore-Plate</td>
<td>300</td>
<td>70</td>
</tr>
<tr>
<td></td>
<td>0.98-Aw NaCl</td>
<td></td>
<td></td>
</tr>
<tr>
<td>9</td>
<td>Fresh WBM/Soltex</td>
<td>300</td>
<td>70</td>
</tr>
<tr>
<td></td>
<td>0.98-Aw NaCl</td>
<td></td>
<td></td>
</tr>
<tr>
<td>10</td>
<td>Fresh WBM</td>
<td>300</td>
<td>240</td>
</tr>
<tr>
<td></td>
<td>0.98-Aw NaCl</td>
<td></td>
<td></td>
</tr>
<tr>
<td>11</td>
<td>Fresh WBM/Bore-Plate</td>
<td>300</td>
<td>240</td>
</tr>
<tr>
<td></td>
<td>0.98-Aw NaCl</td>
<td></td>
<td></td>
</tr>
<tr>
<td>12</td>
<td>Fresh WBM/Soltex</td>
<td>300</td>
<td>240</td>
</tr>
<tr>
<td></td>
<td>0.98-Aw NaCl</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
4.5 Test Analysis

As mentioned before, the ability of asphaltic additives in permeability reduction is to be measured. The belief that asphaltic additives physically coat the surface of the shale or extrude in to the micro-pores of the shale thereby blocking filtrate invasion, needs to be verified. A value of permeability called base reference permeability \(K_m\) is measured, as described below, for the experiment (shale) with base mud as the top test fluid. A value of permeability \(K\) is then recorded for experiments (shales) with top test fluid containing Bore-plate or Soltex. A comparison of \(K\) with \(K_m\) will give a quantitative evaluation of permeability change.

**Permeability Calculation**

**Assumption:** There are only two forces driving the flow of filtrate through the shale, Mechanical and Osmotic. The mechanical drive is from the pressure difference across the shale and the osmotic potential due to the difference in water activity’s.

The modified Darcy’s equation can be applied to calculate shale permeability as follows:

\[
k = \frac{245q\mu L}{A(\Delta P_h + \Delta P_{os})}
\]

--------- (4.1)

where, \(k\) = Shale permeability (mD)

\(q\) = Steady-state drainage rate (invasion rate) obtained from the filtration test (ml/min)

\(\mu\) = Water viscosity at test temperature (cp)

\(L\) = Thickness of the shale sample (cm)

\(A\) = Cross sectional area of the shale sample (cm\(^2\))

\(\Delta P_h\) = Hydraulic differential pressure (psi)

\(\Delta P_{os}\) = Osmotic differential pressure (psi)

\((245)\) = Unit conversion factor
As shown in Table 4.2, the top test fluid was a fresh water-based mud (water activity = 1) and the bottom pore fluid was the salt solution at the same water activity as the shale water activity, which was 0.85. Therefore, the osmotic driving force was in the direction from the top fluid to the shale sample. This osmotic differential pressure can be calculated as follows:

\[
\Delta P_{os} = \alpha \left( \frac{RT}{V} \ln \left( \frac{a_s}{a_m} \right) \right) \quad \text{---------- (4.2)}
\]

where, \(\alpha\) = Shale membrane efficiency

\(R\) = Gas constant = 10.73 (psi-ft\(^3\)-lbmol\(^{-1}\)-R\(^{-1}\))

\(T\) = Absolute test temperature (R)

\(V\) = Molar volume of water (ft\(^3\)-lbmol\(^{-1}\))

\(a_m\) = Water activity of mud

\(a_s\) = Water activity of shale

This equation was based on the assumption in which the shale membrane efficiency was unchanged after exposing to the top test fluid.

After the permeability was obtained for each test, the percentage change in permeability was calculated as follows:

\[
\% \text{ Permeability Change} = \left( \frac{k - k_m}{k_m} \right) \times 100 \quad \text{---------- (4.3)}
\]

where,

\(k\) = Shale permeability from the filtration test (mD)

\(k_m\) = Shale permeability from the filtration test with the referenced water-based mud at the same conditions (mD)
4.6 Test Procedure

4.6.1 Preparing the filtration cell

Place the “base” of the cell, with the bolts passed through it, on the workbench. Then carefully position the two layers of wire mesh in to the base. Next place an O-ring in to the groove. Then place the shale sample over the wire mesh and another two layers of wire mesh over the shale sample, perfectly centered. Another O-ring is then placed over the wire mesh and then the “bleed plate” is centered over the O-ring. The “body” of the cell is then carefully slipped through the vertical bolts and on top of the bleed plate. Another O-ring is placed between the body and the bleed-plate in the groove provided on the lower side of the body. The necessary type of mud is then filled in the space inside the body. An O-ring is placed in the groove of the “cap”; the cap is then slipped through the bolts and into the body. The nuts are then tightened to the bolts to a set torque limit of 30 N-m.

4.6.2 Preparing for testing (referred to figure 3.4)

Connect the top pressure gas inlet stem and the bottom outlet stem with the testing system manifold as shown in Figure 3.2. Tighten all connections completely. Set the hand pump at the middle scale (at 12.5 ml). In the case of elevated temperature testing, place the thermocouple electrode inside the oven.

Close all valves and prepare for evacuating air out of the system and filling up the bottom reservoir with the simulated pore fluid.

Open $V_7$ and operate the vacuum pump. Evacuate air until no bubbles come out from the suction tube.

Open $V_6$ and evacuate air until no bubbles come out from the suction tube.
Follow with V_5 and V_4, respectively. Open each valve and evacuate all air before opening the next valve.

Close valve V_7 and shut-off the vacuum pump.

Open the pore fluid source to the atmosphere and let the pore fluid fill up the bottom reservoir.

Close V_6.

4.6.3 Conducting the filtration test

Open the gas valve on the nitrogen gas cylinder. Slowly turn the throttle on the valve till the pressure gauge on the cylinder reads around 300 psi. Open V_2.

Be sure V_3 is closed. When the top fluid pressure reaches 300 psig, pressurize the bottom reservoir (G_3) with the hand pump keeping the differential pressure at 300 psig. However, if the bottom pressure starts to increase due to filtration, releasing the bottom pressure may be required. Finally, the top fluid pressure should be at 500 psig and the bottom reservoir pressure at 200 psig. This bottom pressure suppresses the remaining air in the system.

4.6.4 Monitoring the filtration test

Monitor the bottom pore pressure on G_3. Release it back to 200 psig every 15 minutes with the hand pump. However, if the pore pressure rises very quickly, release bottom pressure to prevent it from exceeding 275 psig. Record the time and volume fluid removed by observing the scale on the hand pump as fluid drainage is in process. From this experiment, the cumulative drainage volume is obtained. Terminate the test when the constant (steady-state) drainage rate (fluid invasion rate) is established.
4.6.5 Conclusion of the filtration test

To end the test, close the valve on the gas cylinder. Then open valve $V_2$ and turn the throttle valve and bleed the rest of the gas until the gas cylinder pressure reads zero.

Open $V_6$ to bleed off pressure from the bottom reservoir and open $V_1$ to bleed off the top fluid pressure.

Disconnect the testing system manifold from the filtration cell.

Dismantle the filtration cell.

4.6.6 Problems faced and measures taken

**LEAKS**

Numerous leaks detected in equipment such as piping, valve seats, joints etc., which were resolved.

**FILTRATION CELL DESIGN**

The original design involved a porous plate between the shale sample and the base of the filtration cell. The purpose of the disk was to channel the filtrate passing through the shale sample, to the downstream side of the experimental set-up and in to the pore-fluid reservoir. But all the porous plates available at our disposal were not perfectly symmetrical in thickness. As a result the groove that housed the porous disk at the base would force itself in to the shale sample consequently cracking it. The only solution to the problem was to machine the base under the shale sample to be flat. And instead of a porous plate, use two layers of wire mesh that would serve the purpose of filtrate channeling.
### Chapter 5: Results and Conclusions

#### TABLE 5.1: Results

<table>
<thead>
<tr>
<th>Shale</th>
<th>Test Number</th>
<th>q (ml/min)</th>
<th>μ (cp)</th>
<th>L (cm)</th>
<th>A (cm²)</th>
<th>k (mD)*10^6</th>
<th>Permeability change %</th>
<th>Temp F</th>
<th>Fluid</th>
</tr>
</thead>
<tbody>
<tr>
<td>Arco china</td>
<td>1</td>
<td>0.00019</td>
<td>1</td>
<td>0.62</td>
<td>20.271</td>
<td>53.95</td>
<td></td>
<td></td>
<td>Base</td>
</tr>
<tr>
<td>Arco china</td>
<td>2</td>
<td>0.00002</td>
<td>1</td>
<td>0.62</td>
<td>20.271</td>
<td>58.33</td>
<td><strong>8.11</strong></td>
<td>70</td>
<td>Bore-plate</td>
</tr>
<tr>
<td>Arco china</td>
<td>3</td>
<td>0.00002</td>
<td>1</td>
<td>0.62</td>
<td>20.271</td>
<td>58.33</td>
<td><strong>8.11</strong></td>
<td>70</td>
<td>Soltex</td>
</tr>
<tr>
<td>Arco china</td>
<td>4</td>
<td>0.00017</td>
<td>0.305</td>
<td>0.62</td>
<td>15.5</td>
<td>19.96</td>
<td></td>
<td></td>
<td>Base</td>
</tr>
<tr>
<td>Arco china</td>
<td>5</td>
<td>0.00017</td>
<td>0.305</td>
<td>0.62</td>
<td>15.5</td>
<td>19.96</td>
<td>0</td>
<td>240</td>
<td>Bore-plate</td>
</tr>
<tr>
<td>Arco china</td>
<td>6</td>
<td>0.00021</td>
<td>0.305</td>
<td>0.62</td>
<td>15.5</td>
<td>28.18</td>
<td><strong>27.54</strong></td>
<td>240</td>
<td>Soltex</td>
</tr>
<tr>
<td>Pierre</td>
<td>7</td>
<td>0.00036</td>
<td>1</td>
<td>0.62</td>
<td>15.5</td>
<td>118.86</td>
<td></td>
<td></td>
<td>Base</td>
</tr>
<tr>
<td>Pierre</td>
<td>8</td>
<td>0.00033</td>
<td>1</td>
<td>0.62</td>
<td>15.5</td>
<td>108.74</td>
<td><strong>-8.52</strong></td>
<td>70</td>
<td>Bore-plate</td>
</tr>
<tr>
<td>Pierre</td>
<td>9</td>
<td>0.00033</td>
<td>1</td>
<td>0.62</td>
<td>15.5</td>
<td>108.74</td>
<td><strong>-8.52</strong></td>
<td>70</td>
<td>Soltex</td>
</tr>
<tr>
<td>Pierre</td>
<td>10</td>
<td>0.00067</td>
<td>0.305</td>
<td>0.62</td>
<td>15.5</td>
<td>132.86</td>
<td></td>
<td></td>
<td>Base</td>
</tr>
<tr>
<td>Pierre</td>
<td>11</td>
<td>0.0005</td>
<td>0.305</td>
<td>0.62</td>
<td>15.5</td>
<td>99.59</td>
<td><strong>-25.04</strong></td>
<td>240</td>
<td>Bore-plate</td>
</tr>
<tr>
<td>Pierre</td>
<td>12</td>
<td>0.0005</td>
<td>0.305</td>
<td>0.62</td>
<td>15.5</td>
<td>99.59</td>
<td><strong>-25.04</strong></td>
<td>240</td>
<td>Soltex</td>
</tr>
</tbody>
</table>
5.1 RESULTS FOR ARCO CHINA SHALE

Test Number 1 evaluated the effect of the referenced water-based mud on the shale sample at ambient temperature. During the test (Fig. 5.1), the top and bottom fluid pressure and temperature were measured and controlled. Therefore, the increase in the bottom pore pressure observed during the filtration test reliably represented fluid invasion into the shale sample. The results produced a steady state filtration rate of 0.00019 ml/min (Fig 5.1) for the base mud (permeability of 53.95 nD, Table 5.1) and a value of 0.0002 ml/min (Fig 5.1) for the additives (permeability of 58.33 nD, Table 5.1).

FIG 5.1: Results of the tests 1,2,3 (Cumulative Drainage Volume Vs Time)
Test Numbers 4, 5, 6 followed the same procedure as test Numbers 1, 2, 3, but the temperature used was 240° F. The filtration rate obtained with the base mud was 0.000167 ml/min (Fig 5.2) and a permeability of 19.96 nD. The additive mud’s, Bore-plate and Soltex, gave comparatively lower initial and steady filtration rates. These mud systems gave a filtration rate of 0.000167 (permeability 19.96 nD, Table 5.1) and 0.000213 (permeability 28.18 nD, Table 5.1) ml/min respectively. The initial stages of filtration in the case of the base mud system hints that a filter-cake was forming (filtration varies linearly with the square root of time).

FIG 5.2: Results of the tests 4, 5, 6 (Cumulative Drainage Volume Vs Time)
5.2 RESULTS FOR PIERRE SHALE

Tests Numbers 7, 8, 9, evaluated Pierre shale at room temperature. A filtration rate of 0.00036 ml/min (permeability 118.86 nD, Table 5.1) was obtained for the base mud system and a rate of 0.00033 ml/min (permeability 108.74 nD, Table 5.1) for the additive systems. Conclusion is that Bore-plate and Soltex system muds have a slightly lower invasion rate, not substantial though. A reduction in filtration rate is a positive indication towards possible permeability reduction; a value of 8.52% was calculated.

FIG 5.3: Results of the tests 7, 8, 9 (Cumulative Drainage Volume Vs Time)
The tests Numbers 10, 11, 12 were performed on Pierre shale at a temperature of 240°F. The base mud gave a filtration rate of 0.00067 ml/min (permeability of 132.86 nD, Table 5.1). The first test on Bore-plate mud system gave a filtration rate of 0.0005 ml/min (permeability 99.59 nD, Table 5.1). The Soltex mud system gave a filtration rate of 0.0005 ml/min (permeability 99.59 nD, Table 5.1). Observations of tests reveal that the Soltex and Bore-plate systems have managed to reduce the filtration rate into the shale by permeability reduction values of 25.04%.

FIG 5.4: Results of the tests 10, 11, 12 (Cumulative Drainage Volume Vs Time)
5.3 CALCULATING THE PERMEABILITY VALUES

As an example, the permeability and the permeability change for test number 6 can be calculated as follows:

Calculation of the osmotic differential pressure, $\Delta P_{os}$

The osmotic pressure differential is assumed to be zero. The reason being, the difference in the water activity’s of the shale and drilling mud induced free transfer of water and ions without hindrance, which implies zero membrane efficiency. Membrane efficiency tests were run to verify this claim and further explanation is presented in the next chapter.

Calculation of the shale permeability, $k$

From the test, $q = 0.000213$ ml/min and $\mu = \text{Water viscosity at } 240 \, ^{\circ}F = 0.305 \, \text{cp}$

$L = 0.25 \, \text{in} = 0.25 \times 2.54 \, \text{cm} = 0.62 \, \text{cm}$

$A = (1.55 \times 1.55^2) = 2.4 \, \text{in}^2 = 15.5 \, \text{cm}^2$

$\Delta P_h = (500-200) = 300 \, \text{psi}$

Applying equation 4.1 to obtain shale permeability ($k$),

$$k = \frac{(245)(0.000213)(0.305)(0.62)}{(15.5)(300)}$$

$$= 28.18 \times 10^{-6} \, \text{mD}$$

Calculation of % permeability change

For this test, shale permeability from test number 4 is the referenced permeability ($k_r$), which is $19.96 \times 10^{-6} \, \text{mD}$.

Therefore, according to equation 4.3, the percent change in permeability equals to:
\[
\% \text{ Permeability Change} = \frac{(28.18 - 19.96) \times 10^{-6}}{19.96 \times 10^{-6}} \times 100 \\
= 27.54\%
\]

5.4 **INTERPRETING THE RESULTS**

The working mechanism of the asphaltic additives is by physically coating the surface of the shale and plugging the shale pores and micro-fractures (it is claimed). The permeability change value (Table 1) answers the question: “*Do Gilsonite and Soltex block filtration into shale?*” If we look at the permeability change expression (equation 4.3) a positive value would indicate that the shale permeability has increased. A negative value of permeability would indicate reduction in permeability. We have to consider the fact that negative osmotic backflow that negates hydraulic flow was not generated and the change in filtration rate is entirely due to the change in permeability. For experiment’s 1 through 6, the permeability change is positive indicating that the additives have not done anything substantial in reducing filtration in to the Arco China shale. For experiments 7 though 12 the permeability change is negative, though not by any means a substantial reduction, for the Pierre shale. It should be noted that the difference in the type of shales could have been the reason for such preferential success. Nevertheless, this is not an excuse because while drilling we can never anticipate the characteristics of the shale to be encountered. At the kind of miniature scales of testing, ideal conditions, excess concentrations and static state (all factors conducive), incapability to deliver satisfactory results is not encouraging.
5.4 CONCLUSION

A limited amount of laboratory tests were performed to evaluate the ability of “Gilsonite” and “Soltex” in shale permeability reduction. The results indicate minimal reduction in permeability of shale samples, which was ‘-6.36%’ for Bore-plate and ‘0.4325%’ for Soltex.

The static filtration test discussed is one of the methods to study the effects of asphaltic coating agents as a potential shale stabilizer. The same equipment and procedure can be used to test other mud formulations, brines and oil based muds.

The working mechanisms of the asphaltic additives are not fully understood and only actual field-testing may prove helpful.

It is recommended that manufacturers of the additives research the reasons behind the results contrary to their claims.

A different water based carrier mud system for the additives may provide efficient sealing.
Appendix A

A.1 UNSUCCESSFUL EXPERIMENTS

The test matrix for unsuccessful experiments has been presented.

TABLE A.1: Test Matrix

<table>
<thead>
<tr>
<th>Test No</th>
<th>Model Example</th>
<th>Differential Pressure (psi)</th>
<th>Temperature (°F)</th>
<th>Shale</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>Top Test Fluid</td>
<td>300</td>
<td>70</td>
<td>Arco china</td>
</tr>
<tr>
<td></td>
<td>Shale</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Bottom Reservoir</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>A</td>
<td>Fresh WBM</td>
<td>300</td>
<td>70</td>
<td>Arco china</td>
</tr>
<tr>
<td></td>
<td>0.85-Aw NaCl</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>B</td>
<td>Fresh WBM/Bore-Plate</td>
<td>300</td>
<td>70</td>
<td>Arco china</td>
</tr>
<tr>
<td></td>
<td>0.85-Aw NaCl</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>C</td>
<td>Fresh WBM/Soltex</td>
<td>300</td>
<td>70</td>
<td>Arco china</td>
</tr>
<tr>
<td></td>
<td>0.85-Aw NaCl</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>D</td>
<td>Fresh WBM</td>
<td>300</td>
<td>240</td>
<td>Arco china</td>
</tr>
<tr>
<td></td>
<td>0.85-Aw NaCl</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>E</td>
<td>Fresh WBM/Bore-Plate</td>
<td>300</td>
<td>240</td>
<td>Arco china</td>
</tr>
<tr>
<td></td>
<td>0.85-Aw NaCl</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>F</td>
<td>Fresh WBM/Soltex</td>
<td>300</td>
<td>240</td>
<td>Arco china</td>
</tr>
<tr>
<td></td>
<td>0.85-Aw NaCl</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>G</td>
<td>Fresh WBM</td>
<td>300</td>
<td>70</td>
<td>Pierre</td>
</tr>
<tr>
<td></td>
<td>0.98-Aw NaCl</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Experiments No. A, B, C were performed with circular shale samples of 2.25” diameter. The mud formulation for the base mud remained the same, while the concentrations of Bore-plate and Soltex were 5 lbm/bbl and 6 lbm/bbl respectively (recommended by their manufacturers). This concentration is one-thirds that of used in all other experiments and for this reason these results were not included. A constant fluid invasion rate was established at 0.0051 ml/min (Fig. A.1) for the base mud. The shale permeability was calculated to be 1487.57 nano-Darcy. This flow rate was used as the reference for Test Numbers 2 and 3 evaluating Bore-Plate and Soltex performance at ambient temperature.

Tests Numbers B & C were performed the same way as test number 1, but had Bore-Plate and Soltex-treated mud as the top test fluid. Again, a stable top and bottom fluid pressure and temperature were observed. This test was to determine if Bore-Plate or Soltex could reduce water penetration under room temperature. Steady fluid invasion rate of 0.0056 ml/min and 0.0156 ml/min (Fig. A.1) were achieved after 90 min for Bore-Plate and 210 min in the case of Soltex, giving permeability values of 1633.41 nD and 4550.22 nD respectively. The permeability value was higher than that obtained from Test No. A (base mud). Neither of the additives provided any permeability reduction under these conditions. It is felt that the higher flow rate reflects sample variation or the presence of micro-fractures.
FIG A.1 Results of the tests A, B, C (Cumulative Drainage Volume Vs Time)

Experiments Nos. D, E, F were performed at 240°F on Arco China shale samples. The additive concentrations were regular and the shale cross-sections were square. The filtration rate obtained with the base mud was 0.02666 ml/min (Fig. A.2) and a permeability of 5382.6 nD. The additive mud’s, Bore-plate and Soltex, gave comparatively higher initial and steady filtration rates. These mud systems gave a filtration rate of 0.0333 ml/min and a shale permeability of 7751 nD. The initial stages of filtration in the case of the base mud system hints that the filter-cake formation-like characteristics (filtration varies linearly with the square root of time) don’t comply. There seems to be some kind of hold-up volume in the equipment. It was found after
performing the experiments that the pressure transducers had drifted away from their calibrated values. This had caused an excessive pressure differential (around 600 psig) to be applied across the shale sample. The results obtained were discarded.

FIG A.2: Results of the tests D, E, F (Cumulative Drainage Volume Vs Time)

Experiment No.G was performed on Pierre shale and Bore-Plate mud system as the top-test fluid at 240°F. The results from this experiment are inaccurate for the same reason as above, drift in the pressure transducer calibration. A steady state filtration rate of 0.001 ml/min (Fig. A.3) was observed and a corresponding shale permeability of 199.19 nD was calculated.
A.2 REPTITION EXPERIMENTS

Three experiments were repeated, exactly similar in nature to Experiment No. 4, 5, 6, from the original test matrix (Table 4.2). The initial filtration characteristics did not match the results obtained, but the steady filtration rate was reproduced exactly. The filtration rate obtained with the base mud was 0.000167 ml/min (Fig A.4) and a permeability of 19.96 nD. The additive mud’s, Bore-plate and Soltex, gave comparatively lower initial and steady filtration rates. These mud systems gave a filtration rate of 0.000167 (permeability 19.96 nD) and 0.000213 (permeability 28.18 nD) ml/min respectively. It can be noted that a few data points (first 120 minutes) for the Bore-Plate
curve are missing. A faulty valve caused problems during the initial part of the experiment.

Fig. A.4: Results for test no. 4, 5, 6, repetition (Cumulative Filtration Vs Time)
Appendix B

B.1 CALIBRATION OF POSITIVE DISPLACEMENT PUMP

The pressure calibration of the positive displacement pump proved that the graduations provided on the device essentially are equal to the filtrate volume numerically. The procedure followed is as follows:

![Schematic for Pump Calibration](image_url)

Fig. B.1: Schematic for Pump Calibration

The positive displacement pump was pressurized to 200 psi and the corresponding reading in ml on the scale on the pump was noted. The valve was then opened and the filtrate sample weight collected in the flask was noted. The next step would be to advance
the pump (valve open) by a notable reading on the pump scale and record the corresponding filtrate volume collected. Thereby the filtrate volume readings from physical observation were compared with that inferred from the pump scale. Both reading were plotted and a slope value of almost 1 was noted (Fig. B.2). The importance of the test is that the steady state filtration rate is read directly from the above pump. Any variation directly affects the permeability calculations.

\[ y = 1.0724x - 0.0673 \]

Fig. B.2: Schematic for Pump Calibration

Also a pressure time plot was plotted (Fig. B.3). The pump was pressurized to approximately 600 psi and the pressure readings recorded by the transducer were recorded with respect to time. With the help of this plot we can interpret the effects of water compressibility hysteresis on measurements.
Fig. B.3: Pressure Vs Time for Positive Displacement Pump
Appendix C

C.1 MEMBRANE EFFICIENCY TESTS

Allowing filtration Experiment No. 1 to continue, without any kind of manual pressure changes applied to the equipment, constituted membrane efficiency test Number 1. The Arco China shale has a water activity of 0.85 and the test calls for a brine of 0.85 as the bottom pore fluid, to match the activity and thereby reduce any osmotic potential. The water activity of the top test fluid (WBM) is almost 1. A very low value of membrane efficiency would be expected due the considerable difference in water activity between the shale sample and the fluid. As expected the test gave a value of 0.4% membrane efficiency. This value was calculated using Equation No. 2.1 and 2.2. The low value justifies our assumption that the filtration tests performed have no osmotic potential involved.

Membrane efficiency of Arco China Shale when exposed to a brine of water activity 0.85

Fig. C.1: Membrane Efficiency Calculation


Vita

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