Effect of Porosity and Permeability on the Membrane Efficiency of Shales

By

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Thesis

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Effect of Porosity and Permeability on the Membrane Efficiency of Shales

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Dedication

I would like to dedicate this thesis to my parents, Mr. and Mrs. U. N. Osuji, whose support, prayers, and encouragement were helpful during this graduate work. I would also like to dedicate it especially to my grandmother, Mrs. Asije Ihelewere nee Babangida, for her tremendous support, love, and affection for me. Also, I dedicate it to my brother, Nnorom, and my sisters, Uche, Linda (for her financial support), Chinwe, Uzoma, and Ihuoma, for providing the family environment that has guided me through life. Finally, I would like to dedicate it to God, who has given me the opportunity, humility, faith, strength, and intelligence to succeed in life.
Acknowledgements

I would like to thank Dr. Martin E. Chenevert and Dr Mukul M. Sharma for their excellent guidance, mentorship, and priceless technical insight throughout this research. I have greatly enjoyed honing my problem solving skills under their able supervision.

I would also love to extend my sincere gratitude to Glen Baum, Don Sorrell, and Tony Bermudez for all their timely and excellent support during this work. Finally, I thank Taner Sensoy for his engaging interest, good humor, and insight during the development of the tests.
This study presents experimental data showing the dependence of shale membrane efficiency on petrophysical properties and mud composition for water-based muds. Wellbore instability often occurs as a result of osmotic pressures that develop when a shale is in contact with water-based muds. The osmotic pressures generated are proportional to the shale membrane efficiency. A pressure transmission technique was used to measure the membrane efficiency of Atoka and C-5 shale at different porosities. It is not currently possible to directly measure the shale membrane efficiency down-hole. This method may provide a way to estimate shale membrane efficiency from down-hole wire-line formation tester measurements. The results of this study could possibly be used to design muds that produce shale strengthening and result in better wellbore stability.

Two series of tests were performed; the first set used brine solution as the test fluid while the second set used three, industry-provided, water-based muds. Results show that the membrane efficiency of Atoka shale ranges from 0.4% to about 13% and is a function of the shale porosity. This shows that the porosity of the shale itself is an important
consideration in mud design. The data clearly show that the membrane efficiency is negatively correlated with the shale porosity until a porosity of about 7.5%, beyond which there is essentially no change in membrane efficiency. A good correlation was also found between the shale permeability (which is in the order of 0.1 nD) and the membrane efficiency. Above a permeability of 0.2 nD, osmotic effects were measured to be small.

For tests conducted with water-based muds, the membrane efficiency of the shale was reduced by a factor of more than 2 after contacting two of the three muds with the mud-altered Atoka shale. This decrease was found to correlate very well with an increase in porosity and permeability in the shale. However, muds that reduced the permeability exhibited an increase in membrane efficiency. This shows the importance of porosity-and permeability-reducing agents in changing the membrane efficiency and osmotic pressure in shales.

Shale membrane efficiency has been shown to correlate with the shale porosity and permeability. Interaction of the shale with different water-based muds is shown to change the membrane properties of the shale. Furthermore, the nature of this change determines the effectiveness of these muds in the stabilization of troublesome shales. This is important because of the time-dependent nature of wellbore failure. This study shows that certain drilling fluids have the ability to alter the shale through permeability reduction induced by osmotic flow. The results of the study can be used to design better water-based drilling fluids that will stabilize shales.
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I. Introduction

Background and Literature Review

Shales are clastic sedimentary rocks with distinct laminated layers containing lithified clay-sized mineral particles. The clay constituents are often characterized by a large surface area and associated bound water. The clay types include smectite, which has water and sodium ions associated with it. Smectite is a swelling clay and has the highest surface area (about 750 m$^2$/gm). With a surface area of about 80 m$^2$/gm, illite is a non-swelling clay and has potassium as the interlayer cation with little or no associated water. Kaolinite has a small degree of isomorphic substitution in its structure and has a very low surface area and cation exchange capacity. The characteristics of any particular shale are often governed by the amount of each clay mineral present. Shales that contain more smectite tend to be very reactive and highly susceptible to swelling.

The peculiar properties of shale make them very troublesome in drilling operations. Also, with current advances in oil-well drilling technology, such as multilateral and extended reach wells, the importance of maintaining a stable wellbore over very long angle build-up sections cannot be overemphasized. Lal (1999) observed that over 75% of the formations drilled worldwide are classified as shale formations, and about 70% of borehole problems during drilling operation are related to shales. Clearly, this shows the importance of shale to wellbore stability. Some of the problems related to shales range from, hole-enlargement, tight-hole, stuck-pipe, high torque and drag, fracturing, well
control, and total wellbore collapse which could result in well abandonment. This is the reason behind the large number of studies to minimize or eliminate shale instability.

Oil-based muds and synthetic muds have been used to combat the problem of shale instability in the wellbore. The high capillary-pressure generated between the oil-based mud and the shale pore fluid is responsible for restricting the inflow of oil filtrate into the shale. Also, the saline internal phase of the mud prevents the movement of water into the shale. However, their use has been restricted world-wide, due to environmental concerns and their high cost of implementation. This has spurred interest among researchers to develop environmentally-friendly water-based muds that would stabilize shales as effectively as oil-based muds.

This study will deal primarily with salt solutions and water-based muds. Several researchers have concluded that when water-based muds are used, there are two basic phenomena that cause shale instability in the wellbore. These are classified into mechanical and physico-chemical effects. Mechanical effects are physical processes that occur when the mechanical stress imposed on the shale by earth stresses and the wellbore exceeds the shale strength. This can be effectively managed by altering the mud weight to keep it within the shale strength window. However, unlike mechanically induced wellbore failure, chemically induced wellbore failures are time-dependent, and are due to stresses and pressures generated within the shale as a result of shale/drilling-fluid interaction.
Also, when drilling overbalance, the mud overbalance pressure penetration will result in pore pressure increase near the wellbore. This is aided by the fact that shales (unlike other formations encountered in wells) generally have very low permeability, which affects the formation of a protective filter cake. However, slow drilling fluid invasion equilibrates the mud and the near-wellbore stress, whereby effective mud pressure support is lost. Shales adjacent to the wellbore may yield in shear or tensile modes because of this pore pressure elevation.

Low and Anderson (1958) presented osmotic pressure equations suggesting osmosis as a mechanism for swelling pressures generated by shales. This principle is based on the fact that the shale acts in conjunction with certain muds as a semi-permeable membrane, which allows for the generation of osmotic pressure between the shale and the drilling fluid. The main driving force is the chemical potential difference between the shale’s pore fluid and the drilling fluid.

Whitworth and Fritz (1994) noted that the fine pore size and the negative charge of clay on pore surfaces cause shales to exhibit a semi-permeable membrane behavior. This has led to efforts to use the osmotic pressure to extract water out of the shale, which will result in the strengthening of the shale around the wellbore. However, shales do not act as a perfect membrane and have been described as leaky. Shales are supposed to restrict the flow of solutes while flow of water is relatively unrestricted when high saline muds are
used. If this happens, the chemical potential differential imposed could drive water out of the shale. However, it has been observed that shales generally allow the passage of some solutes and water, thereby altering the magnitude of the osmotic potential. In the case of shales with higher permeability, convection could override osmosis, thereby equalizing the chemical potential in the wellbore and the shale formation. This could leave the near wellbore shale susceptible to the hydraulic pressure overbalance imposed by the mud.

Clearly overcoming shale instability, when using water-based muds, is a significant challenge. Several studies have been conducted on the transport of ions and water through shales and its impact on shale stability. However, factors that control the membrane efficiency still need to be fully explored and understood. This would help in the design and successful implementation of water-based fluid systems in drilling operations. The study contained herein seeks to understand the petrophysical changes that occur in shales when they come into contact with water-base fluid systems. Also, the effects of porosity and permeability on the membrane efficiency of shale were studied and relevant correlations between these properties were explored. Hopefully, this will provide a way to estimate the membrane efficiency of shales from wire-line measurements.
Problem Formulation

Shale instability is primarily governed by the hydraulic and chemical interaction between the shale and the drilling fluid. Drilling fluids often induce failure by changing the stress state of the near wellbore formation through this shale - drilling fluid interaction. The main driving force for this interaction is the difference in chemical potential between the shale’s pore fluid and the drilling fluid. Most water-based muds use their chemistry and mud weight to control the flow of ions and water in and out of the near wellbore region, and thereby control the hydration stress, pore pressure, and the resulting shale strength.

The level of success in controlling the flow of water and ions through the shale is measured by the shale membrane efficiency. Researchers such as Van Oort et al. (1996), Ewy and Stankovich (2000), and Al-Bazali et al. (2006) have reported that the membrane efficiency of shale is in the range of 1 to 10%. However, the reasons for this low membrane efficiency have not been fully explored and understood. It is believed that the shale petrophysical properties determine the shale membrane performance. This study will seek to develop a relevant relationship between the shale porosity, permeability, and membrane efficiency.
Scope of Research

The objective of this study was to understand the petrophysical changes that occur in shales during their interaction with water-based muds and their relation to the shale membrane efficiency. I also intended to study such interactions with shales of varying native activity. For this purpose, the Atoka shale and C-5 shale were tested with brine solution and three industry-produced water-based muds.
II. Shale Properties

Introduction

In order to properly characterize and understand the results of the experimental study, a good understanding of the properties of the shale studied is necessary. Some of the properties that characterize shales are native moisture content, native water activity, cation exchange capacity, and the clay content. These are important properties for proper interpretation of the test results. Also, due to the susceptibility of shale cores to rapid deterioration when exposed to the atmosphere, core field samples used in this study were preserved using several layers of polyethylene bags and plastic coating; this ensured that the shale was tested in conditions that most represent their native state. However, during this study, the shale was hydrated to different water activity levels, in order to study the effect of porosity on membrane efficiency. The mineralogical analysis of the shale used in this study was performed by OGS Laboratory.

Mineralogical Composition

Two shales are reported on in this work: the hard Atoka and the soft C-5 shale. The Atoka shale has a relatively high quartz content (about 52 %) and a low water activity. Based on the X-ray diffraction analysis performed by Newpark Drilling Fluids, kaolinite and illite appear to be the main clays present, constituting more than 63% of the total clay content. Smectite, which has the highest tendency to absorb water and swell, is about 20% of the clay content, and therefore only 6.3% of the Atoka shale.
Table 1: Mineralogical composition of Atoka shale

<table>
<thead>
<tr>
<th>X-ray Diffraction</th>
<th>Wt (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Quartz</td>
<td>52</td>
</tr>
<tr>
<td>Feldspar</td>
<td>15</td>
</tr>
<tr>
<td>Total Clay</td>
<td>33</td>
</tr>
<tr>
<td>Kaolinite</td>
<td>32</td>
</tr>
<tr>
<td>Chlorite</td>
<td>7</td>
</tr>
<tr>
<td>Illite</td>
<td>31</td>
</tr>
<tr>
<td>Smectite</td>
<td>19</td>
</tr>
<tr>
<td>Mixed Layer</td>
<td>11</td>
</tr>
<tr>
<td>Illite/smectite</td>
<td>19/81</td>
</tr>
</tbody>
</table>

The Cation Exchange Capacity (CEC) of Atoka shale is 13.5 meq/100g, which suggests that the shale is not very reactive. Such low reactivity is confirmed by its adsorption/desorption isotherm.

Atoka Shale Cation Exchange Capacity by Cation Type

Table 2: Exchangeable bases in Atoka shale

<table>
<thead>
<tr>
<th>Cations</th>
<th>meq/100g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sodium</td>
<td>3.6</td>
</tr>
<tr>
<td>Potassium</td>
<td>1.2</td>
</tr>
<tr>
<td>Magnesium</td>
<td>1.2</td>
</tr>
<tr>
<td>Calcium</td>
<td>4.8</td>
</tr>
</tbody>
</table>

The CEC of the softer C-5 shale, supplied by Chevron, is 27 meq/100g. This suggests that the C-5 is more reactive than the Atoka shale. Also, C-5 shale has a quartz content of about 32%, which is lower than that of Atoka shale. The clay content is higher than that
of Atoka shale and constitutes 44% of the total C-5 weight, as shown in the mineralogical data shown in Table 3.

Table 3: Mineralogical composition of C-5 shale

<table>
<thead>
<tr>
<th>X-Ray Diffraction</th>
<th>Wt (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Quartz</td>
<td>32%</td>
</tr>
<tr>
<td>Feldspar</td>
<td>24%</td>
</tr>
<tr>
<td>Total Clay</td>
<td>44%</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Clays</th>
<th>Wt %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Illite</td>
<td>13%</td>
</tr>
<tr>
<td>Smectite</td>
<td>7%</td>
</tr>
<tr>
<td>Kaolinite</td>
<td>50%</td>
</tr>
<tr>
<td>Chlorite</td>
<td>1%</td>
</tr>
<tr>
<td>mixed layer</td>
<td>29%</td>
</tr>
</tbody>
</table>

**Native Water Activity and Native Moisture Content of Atoka Shale**

The water activity of shale is defined as the vapor pressure of the shale divided by the vapor pressure of pure water at the same temperature. The native moisture content is the amount of water contained within the shale’s pores, in its native state. It is evident that the moisture content and native water activity are closely related. Therefore, following the procedure developed by Chenevert (1970), the adsorption isotherms for the Atoka shale and the C-5 shale were measured to determine their native activity.
Native Activity of Shale

The procedure adopted for determining the native water activity includes cutting four shale samples into dimensions 0.5” x 0.5” x 0.5” using a 6” circular saw. These samples were immediately weighed to determine their native weight. They were then placed into desiccators with relative humidities (water activity) of 0.3, 0.54, 0.86, and 0.98. Their weights were recorded daily, showing that some of them gained weight while others lost weight. At equilibrium, when the samples neither gain nor lose weight, the amount of water gained or lost from the initial weight is recorded. The percentage of weight gained or lost is plotted against the relative humidity (water activity) of the desiccator in which they achieved equilibrium. This plot is the adsorption/desorption isotherm and it is shown in Fig. 1 for the Atoka shale and in Fig. 2 for C-5 shale.
The native activity of the Atoka shale is the activity at which it is neither gaining nor losing water, i.e. at a gain of 0%, which is at a relative humidity (water activity) of 0.76. This is the native activity of the Atoka shale used in this study.
The desorption/adsorption isotherm for C-5 shale confirms that it is a relatively soft shale with a native activity of about 0.998.
For comparison purposes, Fig. 3 presents adsorption isotherms of different shales alongside those of the Atoka and the C-5 shales. As mentioned in the article by Chenevert (1970), shale characteristic adsorption isotherms can be used to measure their reactivity. The shales that plot higher in the chart are more reactive than those that plot lower.

Fig. 3: Comparison of Shale Adsorption Isotherms; data is culled from (Chenevert, 1970)
Native Moisture Content

The native moisture content of the shales was determined by cutting seven cubic samples (0.5” x 0.5” x 0.5”) of native shale, using an oil cooled 6” circular saw. After cutting the shale samples, they were cleaned of Escaid mineral oil (in which they were initially stored) using hexane. Then their initial weight was recorded. The samples were then placed in an oven, maintained at 220°F, and their weight measurements were taken daily until there was no measurable weight change (72 hrs was sufficient in this case). The dry weights of the samples were then recorded.

\[
\text{Native Moisture Content (\%)} = \frac{W_{\text{native}} - W_{\text{dry}}}{W_{\text{native}}} \times 100
\]

Table 4: Native Moisture Content of Atoka shale

<table>
<thead>
<tr>
<th>Sample</th>
<th>Initial weight</th>
<th>72hr Dry weight</th>
<th>Native Moisture Content (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>7</td>
<td>5.6565</td>
<td>5.4713</td>
<td>3.3849</td>
</tr>
<tr>
<td>8</td>
<td>5.5585</td>
<td>5.3764</td>
<td>3.3870</td>
</tr>
<tr>
<td>9</td>
<td>7.1559</td>
<td>6.9505</td>
<td>2.9552</td>
</tr>
<tr>
<td>11</td>
<td>6.3405</td>
<td>6.1281</td>
<td>3.4660</td>
</tr>
<tr>
<td>12</td>
<td>5.6999</td>
<td>5.5125</td>
<td>3.3995</td>
</tr>
<tr>
<td>10</td>
<td>5.8644</td>
<td>5.6905</td>
<td>3.0560</td>
</tr>
<tr>
<td>13</td>
<td>4.2231</td>
<td>4.0987</td>
<td>3.0351</td>
</tr>
</tbody>
</table>

\[ \text{Native Moisture Content} = 3.24 \]

Table 5: Native Moisture Content of C-5 shale

<table>
<thead>
<tr>
<th>Sample</th>
<th>Initial weight</th>
<th>72hr Dry weight</th>
<th>Native Moisture Content (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2</td>
<td>17.5</td>
<td>15.4</td>
<td>13.7191</td>
</tr>
<tr>
<td>3</td>
<td>13.9</td>
<td>12.2</td>
<td>13.7188</td>
</tr>
<tr>
<td>4</td>
<td>8.3</td>
<td>7.3</td>
<td>14.0373</td>
</tr>
<tr>
<td>5</td>
<td>13.1</td>
<td>11.5</td>
<td>13.6078</td>
</tr>
</tbody>
</table>

\[ \text{Native Moisture Content} = 13.77 \]
**Relationship between Shale Activity and Porosity**

Since this experimental study intends to show the relationship between porosity, permeability, and membrane efficiency of shale, it is important that the relationship between shale activity and shale porosity be determined. This was done by altering the water content of various shale samples by changing their water activities in various desiccators. Then, the porosity of the samples was determined.

By definition, porosity of a material is equal to the volume of the pore space divided by the bulk volume of the material. In our tests, the volume of pore space was assumed to be equal to the volume of water contained in the sample. Thus, the pore volume could easily be determined by weighing the sample before and after drying, and dividing the weight loss by the density of water. The resultant quotient was assumed to be the pore volume.

The bulk volume was determined by two methods, referred to herein as the Caliper method and the Weight method.

In the *Caliper method*, the bulk volume was calculated by measuring the dimensions of the cubic samples using a Cen-Tech™ digital caliper, accurate to 0.001 inch. Then, the sides were multiplied to calculate the bulk volume \( V_{\text{bulk}} \).
In the *Weight method*, knowledge of the samples’ dimensions is not required to calculate the bulk volume. However, the grain density is required. Using an assumed grain density of 2.7 gm/cc, the bulk volume of a given sample was calculated using equation 2.

\[
V_{\text{bulk}} = \frac{W_w}{\rho_w} + \frac{W_{\text{grain}}}{\rho_{\text{grain}}} \tag{2}
\]

Where \( W_w \) is the weight of water lost or gained, and is equal to \( W_{\text{final}} - W_{\text{dry}} \).

\[
\text{Porosity}(\phi) = \frac{V_w}{V_{\text{bulk}}} \cdot 100 \tag{3}
\]

![Graph showing Atoka shale porosity as a function of its water activity](Fig. 4)

**Fig. 4**: Atoka shale porosity as a function of its water activity

Figures 4 and 5 presents the results of the two methods. As shown in Fig. 4, good agreement between the two methods was obtained for Atoka shale.
The Caliper and Weight method agree quite well for water activities above 0.92, as shown in Fig. 5. The departure in porosity for activities of 0.9 and 0.86 suggests that the bulk volume obtained by the Caliper method was too high, possibly due to air entering the samples. If so, those two samples would be unsaturated.

The figures above capture the relationship between shale porosity and shale activity. As expected, high porosity reflects high shale activity. Shale activity is a measure of the amount of water present in the shale pores at a given aqueous chemical state. The shale’s native activity state is an indication of the amount of water it contains in-situ and is, therefore, related to its in-situ porosity. It is believed that porosity plays a major role in determining the membrane behavior of shale, as well as the shale stabilization technique employed in designing drilling fluids.

**Fig. 5:** C-5 shale porosity as a function of its water activity
Shale Saturation Tests

Shales cored from wells are known to be susceptible to deterioration if exposed to the atmosphere. All attempts to investigate the characteristics of any particular shale from such cores would be questionable if the cores are unsaturated.

Chenervert and Amanullah (2001) developed an immersion technique to determine the saturated condition of shale core samples before using them in any experimental study. The technique consists of first, changing the water content of the shale, then second, determining the resulting volume change by two independent methods: an oil-immersion method and a weight change method. If the two changes do not agree, then it is probable that air has entered the shale and thus, the shale is no longer saturated.

The tests involved cutting small samples of native shale (1”x0.5”x0.5”), recording their weights ($W_i$), and then placing them in various hydrating (or dehydrating) desiccators. The native bulk volume ($V_{nb}$), which is the bulk volume before hydrating, was determined by weighing the native sample in air and weighing it immersed in mineral oil.

$$V_{nb} = \frac{W_i - W_{im1}}{\rho_{oil}}$$

where $W_i$ is the native weight in air and $W_{im1}$ is the immersed weight of the native sample. After the desiccated samples had reached equilibrium, a sample was removed from a desiccator, quickly weighed in air ($W_{final}$), and then immersed in oil and again weighed ($W_{im2}$).
The bulk volume after equilibrium in the desiccator was recorded as

\[ V_{ba} = \frac{W_{\text{final}} - W_{\text{im2}}}{\rho_w} \]

where \( W_{\text{final}} \) and \( W_{\text{im2}} \) are the respective weights of the sample in air and immersed in oil, after achieving equilibrium in the desiccators. The sample bulk volume change by the immersion method was determined using Equation 4,

\[ \Delta V_{bi} = V_{ba} - V_{nb} \]

Next, the bulk volume change by the weight method was determined using equation 5,

\[ \Delta V_{bw} = \frac{W_{f\text{inal}} - W_i}{\gamma_w} \]

This process was repeated for samples placed in other desiccators and the results were plotted as shown in Figs. 6 and 7.

With this method, any values not falling on the 45 degree line imply that water desorption or absorption by the shale does not correspond to a change in bulk volume, which further implies the existence of air spaces inside the shale. This would lead to erroneous results in studies using such unsaturated shale samples.
Data for all four points of Fig. 6 fall on the 45° line, thus the Atoka shale samples were considered saturated. However the C-5 data did not fall on the 45° line, showing that the sample might be under-saturated at these activities.

Fig. 6: Saturation test for Atoka shale

Fig. 7: Saturation test for C-5 shale
III. Experimental System and Components

**Main Testing System**

The pressure transmission technique was used in this study to measure the porosity, permeability, and membrane efficiency of shale samples. The technique consisted of flowing the test-fluid across the top of the sample at a constant pressure (about 250 psi) and comparing it to the bottom pressure as the test proceeded. The bottom fluid consisted of a low salinity NaCl solution (\(a_w=0.98\)) intended to simulate the fluid in the pores of the shale. At the initiation of the test, the bottom pressure was set at about 50 psi, which then increased, or decreased, depending on the membrane efficiency of the shale, to the pressure of the top test-fluid.

In the case where the bottom fluid pressure increased to that of the top fluid, it was concluded that the system had zero membrane efficiency. For a mud-shale system to have 100% membrane efficiency, the final differential pressure between the top and bottom chamber must match the calculated theoretical value. However, if the final pressure differential does not reach the calculated theoretical pressure, the membrane efficiency will be less than 100%.
The test cell used in this test is shown in Fig. 8. This cell, where the shale sample was held, consisted of stainless steel top and bottom caps, attached together with 0.25" grade 3 bolts. The volume of the bottom chamber was kept very small by connecting a “zero volume” pressure transducer to it. This was done so as to ensure an accurate and rapid recording of the simulated pore fluid response. Also, the cell had grooves for two 2.5” x 0.228” O-rings that provided an effective pressure seal for the top and bottom caps. Each side of the shale was fitted with three layers of screen wire to allow for flow across the face of the shale and help apply a uniform distribution of mechanical stress across the surface area of the shale sample. The top side of the sample was lined with two 117 by 117 mesh.
screens and one 14 x 18 coarse screen. Since the bottom fluid was clear brine, there was no chance of it plugging the lines. Therefore, it was fitted with three 117 by 117 mesh screens. A picture of the assembled cell is shown in Fig. 9.

![Fig. 9: Coupled Test Cell](image)
The hydraulic experimental set up is shown in Fig.10. The whole system consists of several devices used to achieve a continuous flow of the test-fluid across the top face of the shale sample, while the simulated pore fluid was kept in contact with the lower face of the shale. A back pressure regulator (BPR) was used to ensure that constant pressure was maintained in the top chamber during the test. Also, a vacuum pump was used to
remove air from the system before running an experiment, thereby minimizing errors due to air compressibility of the fluids.

Other components include:

**Downstream Manual Pump**

The 25 ml- capacity downstream piston-type pump was used to manually insert the simulated pore fluid pressure into the lower chamber of the test cell.

**Test Fluid Injection Pump**

An injection pump was used to circulate the test-fluids across the surface of the shale at a constant rate. This was achieved by pumping water against one side of the floating piston of the test-fluid cylinder that contains test-fluid on the other side.

**Nitrogen Cylinder**

The nitrogen cylinder was used to supply nitrogen to the back pressure regulator.

Other equipment included a Fisher® Isotherm Vacuum Oven, used to dry the samples while determining their native moisture content and porosity. A Digi-Sense® Thermo hygrometer was used in determining the water activity of the different muds used in the experiment.
Figure 11 shows the complete system set up inside an oven. To the left of the test cell is the 158 ml test fluid cylinder containing the fluid. Several pressure transducers are shown at appropriate positions in the manifold. They continually transmit pressure measurements to the recorder.
Shale Sample Preparation

When preparing a test specimen, the following steps were taken. First, the whole diameter core was removed from the can of Escaid oil, then cut into 1.3” x 1.3” x 6” long columns. These columns were then placed in a 2.5” ID acrylic plastic tube and the end was cemented onto a 1.4”x1.4”x 0.25” thick acrylic base using a 5 minute 2-part epoxy, as shown in Fig. 12.

Fig. 12: Preparation of the Shale samples; Prior to epoxy pouring

A 50/50 mixture of epoxy resin and its curing agent was then poured into the tube, and a 2” ID x 4” long sand stone piece was inserted on top of the shale to facilitate handling. The sample was allowed to set for about 7 days. It was then sliced into 0.275” thick samples using an oil cooled diamond circular saw. After slicing, the acrylic plastic ring

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(from the acrylic plastic tube) was removed from the hardened epoxy, leaving 2.5” ID x 0.275” thick disk-like samples, as shown in Fig 14. The samples were immediately immersed in a can containing Escaid mineral oil.

By removing the acrylic ring, leakage between the acrylic ring and the epoxy was eliminated. During testing, the shale sample was held firmly by the O-rings, sealing against the epoxy.

![Fully cured epoxy and shale sample inside acrylic plastic tube](image)

**Fig. 13:** Fully cured epoxy and shale sample inside acrylic plastic tube
**Fluid Types and Properties**

**NaCl Solution**

Experiments were performed using one salt water fluid and three typical drilling muds. In order to investigate the mechanism and characteristics of solute transport involved in the osmotic interaction of salt solution and shale, preliminary pressure transmission tests were conducted using a 0.85 $a_w$ NaCl solution as the upstream fluid, flowing across the top of the shale at an elevated pressure of about 300 psi, and a stagnant 0.98 $a_w$ NaCl solution, at about 50 psi on the bottom side. It was determined that if the fluid-shale system had a reasonable membrane efficiency, the water activity difference between the top and bottom fluid activities was sufficient to overcome the 250 psi hydraulic differential, and also drive water from the bottom to the top of the cell. Because the bottom chamber is very small, this should result in the continual decrease of the bottom chamber pressure. However, a combination of the shale petrophysical properties and the reaction of the shale with the fluid will determine the real evolution of the bottom
chamber pressure. In this test, as well as others, when simulated pore fluid was used, it was mixed using de-ionized water, so as to ensure accuracy.

**Mud Properties**

Three water-based muds were submitted by various mud companies and used in this test program. They were first filtered using a 117 by 117 screen to remove any solids that might plug the lines during the experiment. The muds were labeled A, B, and C and their compositions are given in Tables 6, 7, and 8. The water activity of the muds was determined using a Digi-Sense Thermo hygrometer. Their values, as well as their pHs, are given in Table 9. During testing, the line pressure would fluctuate with changes in temperature. This is why the experiments were conducted in a closed oven, capable of maintaining a constant temperature throughout the experiment.

**Table 6: Mud Composition for Mud A**

<table>
<thead>
<tr>
<th>Mud A</th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Water</td>
<td>0.69</td>
<td>bbl</td>
</tr>
<tr>
<td>Biopolymer</td>
<td>2.25</td>
<td>ppb</td>
</tr>
<tr>
<td>Blend of Polyglycerines</td>
<td>20</td>
<td>% by Vol</td>
</tr>
<tr>
<td>Modified Ester</td>
<td>5</td>
<td>% by Vol</td>
</tr>
<tr>
<td>High Grade Leonardite</td>
<td>8</td>
<td>ppb</td>
</tr>
<tr>
<td>Potassium silicate shale stabilizer</td>
<td>4</td>
<td>ppb</td>
</tr>
<tr>
<td>Potassium Carbonate (K₂CO₃)</td>
<td>4</td>
<td>ppb</td>
</tr>
<tr>
<td>KCl</td>
<td>75</td>
<td>ppb</td>
</tr>
</tbody>
</table>
### Table 7: Mud composition for Mud B

<table>
<thead>
<tr>
<th>Ingredient</th>
<th>Quantity</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water</td>
<td>0.87 bbl</td>
</tr>
<tr>
<td>NaCl</td>
<td>76 lb</td>
</tr>
<tr>
<td>Powdered, high-molecular weight partially hydrolyzed polyacrylamide</td>
<td>0.4 lb</td>
</tr>
<tr>
<td>Low viscosity polyanionic cellulose</td>
<td>2 lb</td>
</tr>
<tr>
<td>Aluminum/resin for shale stability</td>
<td>4 lb</td>
</tr>
<tr>
<td>Deformable sealing polymer</td>
<td>3 %</td>
</tr>
<tr>
<td>Clay Hydration Suppressant</td>
<td>7 lb</td>
</tr>
<tr>
<td>NaOH</td>
<td>0.3 lb</td>
</tr>
</tbody>
</table>

### Table 8: Mud Composition for Mud C

<table>
<thead>
<tr>
<th>Ingredient</th>
<th>Quantity</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water</td>
<td>0.88 bbl</td>
</tr>
<tr>
<td>NaCl</td>
<td>120 lb</td>
</tr>
<tr>
<td>Clay Inhibitor</td>
<td>2 lb</td>
</tr>
<tr>
<td>Clay Inhibitor</td>
<td>1.5 lb</td>
</tr>
<tr>
<td>Liquid Inorganic salt blend</td>
<td>0.02 bbl</td>
</tr>
<tr>
<td>Low viscosity polyanionic cellulose</td>
<td>2 bbl</td>
</tr>
<tr>
<td>Modified Starch</td>
<td>1 lb</td>
</tr>
<tr>
<td>Premium Dispersion Enhanced Xantham</td>
<td>1 lb</td>
</tr>
<tr>
<td>Polyalkylene Glycol</td>
<td>0.02 bbl</td>
</tr>
</tbody>
</table>

### Table 9: Water Activity and pH measurements for the muds used

<table>
<thead>
<tr>
<th>Mud</th>
<th>Water Activity</th>
<th>pH</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mud A</td>
<td>81.4 %</td>
<td>10.5</td>
</tr>
<tr>
<td>Mud C</td>
<td>81.0 %</td>
<td>8.6</td>
</tr>
<tr>
<td>Mud B</td>
<td>84.5 %</td>
<td>9.7</td>
</tr>
</tbody>
</table>
IV. Test Description and Procedure

Test Procedure

The test device and procedure used is similar to the membrane efficiency test device previously used in our laboratory by Al-Bazali et al. (2006). However, the procedure and equipment have been upgraded for a more accurate measurement. In particular, a “zero volume” transducer, supplied by Honeywell Sensotec Sensors, was attached to the bottom cap of the cell, allowing for more rapid response. As a result, a five day test has been reduced to 24 hours.

Before the sample was used, the mineral oil was washed off the sample using hexane. The sample was then placed in a relative humidity-controlled desiccator that has a known water activity, and the sample was allowed to reach equilibrium. This was done by taking daily weight measurements until there was no change in weight. After the shale sample reached equilibrium, it was ready for testing. Before testing began, the top fluid was poured into the mud cylinder and the cylinder was connected to the cell. Then, the 0.275” thick sample was installed into the test cell as rapidly as possible, so as to minimize exposure to the atmosphere. The system was then connected to the injection pump and the manual pump. The vacuum pump was then used to remove air from the lines.

A brine solution of 0.98 water activity was then introduced into the bottom chamber at atmospheric pressure. The top lines and chamber were then filled with the top fluid, using the injection pump at about 15 ml/hr. After circulation was established and confirmed
through the waste outlet, the bottom chamber was pressured to about 50 psi, using the manual pump. The top pressure was then gradually increased to about 300 psi, using the back pressure regulator. After the top pressure reached the desired pressure, the injection pump speed was reduced to about 3ml/hr and valve V2 was closed, so as to confine the 1.13 ml bottom chamber. The experiment was deemed to have started at this stage and the bottom and top pressures were electronically recorded. The test was terminated after the bottom pressure reached a constant value.

**Test Definition and Objectives**

The main objective of this portion of the study was to investigate the qualitative relationship between the Atoka shale petrophysical properties and membrane efficiencies. The shale test samples were hydrated to water activities of 0.76, 0.86, 0.92, and 0.98. These tests were designed to show the impact of shale porosity and permeability on the membrane efficiency. The test imposed both hydraulic and osmotic pressure across the shale and allowed the simulated pore pressure to react to these pressures. The final pressure differential across the shale at equilibrium is converted to membrane efficiency using the following equation.

\[
ME = \frac{\Delta P}{\Pi} \tag{6}
\]

\[
\Pi = \frac{RT}{V} \ln \left( \frac{aw_{df}}{aw_{sh}} \right) \tag{7}
\]
ME = membrane efficiency, (fraction)
Π = the ideal osmotic pressure (for a perfect membrane)
\( aw_{df} \) = the water activity of the mud or drilling fluid
\( aw_{sh} \) = the water activity of the simulated pore fluid
R = universal gas constant \((8.21 \times 10^{-5} \text{ m}^3\text{atm}/\text{Kmol})\)
T = Temperature in absolute units \((°K)\)
V = partial molar volume of water \((18 \text{ cm}^3/\text{mol})\)

where \( ∆P \) is the pressure differential across the shale after equilibrium has been achieved. Also, \( ∆P \) is a function of the shale characteristics and the interaction of the shale pore fluid and the drilling fluid flowing across it.

**Calculation of Shale Permeability**

Shale permeability is an important property in this study because it measures the ability of shale to act as a semi-permeable membrane by restricting the flow of ions through shale. This ability to act as semi-permeable membranes is the result of the low permeability and the negative charge on shale surfaces. Their low permeability is a result of their fine pore size, which enables them to selectively restrict the flow of some ions while allowing the flow of others. Also, differences in the crystal structure of clay sheets, caused by the isomorphic substitution of Al\(^{3+}\) for Si\(^{4+}\) and Mg\(^{2+}\) for Al\(^{3+}\) in their tetrahedral and octahedral sheet, causes shales to have a net negative charge on their surfaces (Schlemmer et al., 2003). This negative charge inhibits the flow of anions and contributes to the membrane behavior of shales. The permeability of the shale during
each experiment was calculated using a transient model developed by Al-Bazali et al. (2006). The derivation is shown in Appendix A.

![Permeability model](image)

**Fig. 15: Permeability model**

The shale permeability ($k$) is calculated as follows:

$$k = \frac{\alpha \mu CVL}{A} \quad \text{................................................................. (8)}$$

Where $\alpha$ is the slope of the graph of

$$ln \left( \frac{P_m - P_{(t,t)}}{P_m - P_o} \right) \text{ vs. time}$$

- $P_m$ is the upstream pressure
- $P_o$ is the initial downstream pressure
- $P_{(t,t)}$ is the downstream pressure as a function of time
- $V$ is the downstream volume i.e. volume of the simulated pore fluid
- $A$ is the shale cross-sectional area
- $C$ is the fluid compressibility
- $\mu$ is the fluid viscosity
- $L$ is the thickness of the shale
V. Test Analysis

Explanation of a Hypothetical Test Result

**Fig. 16**: Test system model

**Fig. 17**: Schematic showing a typical test result
Hypothetical Test 1

In this test, we assume the top fluid to have a water activity of 0.85 and a hydraulic pressure of 300psi. Also, the bottom fluid is assumed to have a water activity of 0.98 and a hydraulic pressure of 50 psi. Given this scenario, there is an osmotic force driving water out of the bottom chamber, through the shale and into the top chamber, as well as a hydraulic pressure driving water from the top to the bottom chamber. The value of the osmotic pressure component is given by,

\[ \Pi = \frac{RT}{V} \ln \left( \frac{A_{W_{df}}}{A_{W_{sh}}} \right) \text{ atm, and it is acting upwards.} \]

For convenience purposes, substituting \(8.21 \times 10^{-5} \text{ m}^3\text{atm}/\text{Kmol}\) for the universal gas constant, \(297 \, ^\circ\text{K}\) for temperature, and \(18 \text{ cm}^3/\text{mol}\) for the partial molar volume of water in the osmotic pressure equation results in

\[ 4.7 \times \frac{RT}{V} = 19,712 \text{ psi.} \quad \text{Thus we have,} \]

\[ \Pi = 19712 \times \ln \left( \frac{0.85}{0.98} \right) = -2805 \text{ psi.} \]

This osmotic pressure component is negative, which means that it is driving fluid upwards. However, there is also a positive hydraulic pressure: \(300 - 50 = 250 \) psi. This pressure is driving fluid downward into the shale. Therefore, the net effect of the hydraulic and osmotic forces is \(-2805 + 250 = -2555\) psia. This means that there is a net pressure of \(2555\) psi driving water from the bottom chamber to the top chamber. If the shale-mud system forms a perfect membrane, the bottom pressure is expected to gradually increase to \(-2555\) psi, i.e. a reduction in \(P_{\text{bottom}}\). This should strengthen the shale, as has been argued by Van Oort et al. (1995).
However, it is observed in Hypothetical Test 1 (as shown in Fig. 17) that the $P_{\text{bottom}}$ gradually increases, instead of decreasing. It finally stabilized at about 200 psi, maintaining an equilibrium $\Delta P_1$ of 50 psi.

Therefore, the membrane efficiency $= \frac{50}{2555} \times 100 = 2\%$

Some have argued that the membrane efficiency is changing as the test proceeds. However, it appears there is also ion movement, in addition to water movement, across the shale. When salt ions move from the top fluid to the bottom fluid through the shale (i.e. the shale is like an imperfect and leaky membrane), the activity difference between the top and the bottom changes. This changing activity difference continues until the activity difference reaches a level where its osmotic pressure can support the hydraulic pressure without a net movement of ions. Also, this can occur when there is a water and/or ionic flux equilibrium across the shale.

Hypothetical Test 1 stabilized at about 50 psi. Theoretically, this implies that there was ion movement into the bottom chamber (i.e. the shale membrane did not perfectly restrict the flow of ions). The shale water activity is no longer 0.98. Instead it has a lower water activity than originally (salt content has probably increased in the bottom chamber).

In order to analyze the membrane behavior of this shale/fluid test, let the new bottom chamber (i.e. pore fluid) activity $= X$.

This now implies that $19,712 \times \ln \left( \frac{0.85}{X} \right)$ (new osmotic pressure) $+ 250$ psi (hydraulic) $= -50$ psi. Back calculating for $X$, which is the new $A_{w_{\text{sh}}}$, $X = \frac{0.85}{\frac{300}{e^{19712}}} = 0.86$. 

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This activity is much lower than the 0.98 simulated pore fluid water activity the experiment started with. This again confirms that the membrane is leaky and there has been ion movement, causing the bottom or pore fluid activity to decrease. Therefore, the osmotic potential required to extract water out of the shale has been reduced.

**Hypothetical Test 2**

In Hypothetical Test 2, again the top fluid activity is 0.85 and the bottom fluid activity is 0.98, and the same net osmotic force of -2555 psi is imposed on the system. As in Test 1, the bottom pressure is expected to decrease until $\Delta P_2 = 2555$ psi.

![Figure 18: Hypothetical Test 2 result](image-url)
The Test 2 pressure profile shows that the bottom pressure did decrease and $\Delta P_2 = 320$ psi. This means that the shale membrane is able to restrict the flow of ions into the bottom chamber more efficiently than in Hypothetical Test 1. However, the fact that $\Delta P_2$ did not decrease to -2555 psi implies that there was some ion transport into the bottom chamber.

Therefore, the membrane efficiency $= \frac{320}{2555} \times 100 = 12.5\%$

The osmotic pressure induced by the new water activity difference, after ion leakage, is still strong enough to drive water out of the bottom chamber. This is why the bottom pressure decreased. The experiments conducted in this study will evaluate the shale properties that control this phenomenon.

**Hypothetical Test 3**

![Graph showing pressure over time for Hypothetical Test 3](image)

**Fig. 19:** Hypothetical Test 3 result
The last scenario (Fig. 19) is a situation in which there is no membrane behavior. The bottom and top pressure will equalize, i.e. $\Delta P_3 = 0$ psi. In this case, there is no restriction to ion movement. The water activity of the bottom and top chambers equalize and there is no osmotic pressure. The hydraulic pressure difference will drive water into the bottom chambers until the pressures equalize.
**System Evaluation Test**

An initial experiment was conducted to evaluate the test procedure and show the osmotic pressure development in our system. The test was conducted by flowing de-ionized water \((a_w = 1.0)\) at 220 psi through the top chamber to the waste outlet and placing 0.98 \(a_w\) brine solution in the bottom chamber at 25psi.

Therefore, there was a hydraulic pressure equal to 220 – 25, or 195psi acting into the shale to increase its pore pressure.

Calculating the osmotic pressure, we have

\[ 19712 \times \ln \left( \frac{1.0}{0.98} \right) = 398 \text{psi} \]

acting across the shale to increase the bottom chamber pressure (i.e. pore pressure).

Therefore, the net effect of the osmotic and hydraulic pressure components is 195 + 398, or 593 psi driving water into the shale to increase the bottom chamber pressure.

![Diagram of the test model](image)

**Fig. 20**: De-ionized water test model

From the configuration of the test, it would be expected that the bottom chamber pressure (containing the simulated pore fluid) would build up immediately because both the...
hydraulic pressure differential and the osmotic pressure generated by the water activity difference between the top and bottom fluid are acting downwards into the shale.

![Graph showing pressure over time](image)

**Fig. 21:** De-ionized water test for system verification

During the test, the bottom pressure was shown to build up to 267 psi, thereby overtaking the top pressure, which was held constant at about 216 psi. However, observe that the bottom pressure did not build up to 593 psi, as expected, but reached a maximum pressure of only 270 psi. This lower pressure suggests that the bottom chamber had been diluted by the de-ionized water to a final activity of about 0.9975. Also, the shale membrane was leaky and did not maintain the chemical potential differential between the top and bottom fluid chambers. It can be inferred that there has been transport of ions that
have unbalanced and reduced the level of osmotic pressure driving water through shale. The bottom pressure profile is what is expected of the shale pore pressure for a field scenario in which shale of 0.98 in-situ activity is drilled with de-ionized water. The pore pressure will increase dramatically and weaken the shale around the wellbore, causing swelling and near wellbore failure. This was represented by the dramatic increase in the bottom pressure beyond the top pressure.
VI. Test Results and Discussion

Test Series 1, Brine Solution Studies

Test Overview

The activities of the Atoka and C-5 shale were altered to different water activities in order to measure the effect of water content on shale porosity, permeability, and membrane efficiency.

In all the tests using brine solutions as the top and bottom fluid, the same osmotic pressure was imposed across the shale. The top fluid was a 0.85 a$_w$ NaCl solution and the bottom fluid was a 0.98 a$_w$ NaCl solution. Given this pressure configuration, the osmotic pressure is shown below

$$\Pi = 19712 \times \ln\left(\frac{0.85}{0.98}\right) = -2805 \text{ psi.}$$

The negative sign shows that an osmotic pressure of -2805 psi acts to extract water out of the shale into the top chamber. In most tests, a hydraulic over balance of about 250psi was used.
Fig. 22: Test 1 for Atoka shale activity =0.98.

Fig. 22 shows the Test 1 results for a sample of Atoka shale that was hydrated to an activity of 0.98 in a controlled desiccator. Due to the osmotic pressure induced on the system by the difference in salt solution activities across the shale, it is expected that this osmotic pressure will act to counterbalance the initial hydraulic pressure differential applied. When this happens, the bottom pressure is expected to continually decrease until the theoretical osmotic differential pressure difference is attained.

However, as Fig. 22 shows, the top pressure is maintained at about 220 psi and the bottom pressure started at about 95 psi. After about 13 hours, the bottom pressure
increased and stabilized at about 200 psi, giving a final pressure differential of 10.8 psi and a resulting membrane efficiency of 0.39 %, as calculated below:

\[
ME = \frac{\Delta P}{\Pi} = \left( \frac{10.8}{2805} \right) \times 100 = 0.39 \%
\]

This is very low membrane efficiency, which shows that there was movement of ions from the lower activity top fluid to the higher activity bottom fluid, because the shale was not restricting ion movement very efficiently.
Example Permeability Calculation for Atoka Shale Test 1

Using the data given in Fig. 22, the plot of Fig. 23 was prepared in order to determine the slope of the curve (α) and thereby calculate the permeability of the shale sample.

\[
y = -0.2807x - 0.0675
\]

\[R^2 = 0.9921\]

**Fig. 23:** Permeability calculation plot

From figure 23, the first 7 hours of data were used to determine the value of \(\alpha\), and equation 8 was used to determine the permeability of the shale.

\[
K = \frac{\alpha q CVL}{A}
\]

where \(\alpha\) is the slope of the graph shown in Fig. 22 which is equal to -0.2807 hr\(^{-1}\)
A permeability of 0.35 nD, which is typical of shales, was calculated (as shown above). It is difficult to control shale instability with a filter cake because, most likely, a filter cake will not form for such low permeability.

![Permeability Calculation Table](image)

Table 10: Example of permeability calculation

![Test 2 for Atoka shale activity =0.92](image)

Fig. 24: Test 2 for Atoka shale activity =0.92

Fig. 24 shows the results for the membrane efficiency test for Atoka shale hydrated to a lower (than?) activity of 0.92. The Atoka shale showed similar behavior as test 1. The
bottom pressure increased from 110psi and stabilized at about 250 psi. The top pressure was maintained at about 275 psi. The lower activity of the shale has a notable effect on the membrane efficiency and the permeability of the shale. At this reduced shale activity of 0.92, the Atoka shale was able to develop a slightly more efficient membrane in order to support about 23 psi pressure across the shale sample. It can be inferred that, at an activity of 0.92, the pore structure and shale lamination geometry are not the same as in a shale of activity 0.98, which is why the mobility of the ions across the shale is lower than at an activity of 0.98, as is observed in test 1. Applying equation 5, the membrane efficiency was determined to be 0.79%.

\[ ME = \frac{\Delta P}{\Pi} = \left( \frac{23}{2805} \right) \times 100 = 0.79 \% \]

The key to enhancing shale stability might be to find a way of reducing the activity of the in-situ shale around the wellbore, as this improves its membrane efficiency and ability to prevent the flow of ions through the shale.
Fig. 25: Test 3 for Atoka shale activity =0.86

Fig. 25 shows the membrane efficiency test results for Atoka shale hydrated to the lower activity of 0.86. It can immediately be observed that the pore pressure (bottom pressure) decreased in this case. Obviously, hydration of the shale to an activity of 0.86 has substantially reduced the movement of ions through the shale. This decrease in pore pressure was achieved because the osmotic differential pressure actually extracted water out of the shale. This would ultimately strengthen the wellbore. Furthermore, this confirms the relationship between the shale activity and the pore throat size, which also defines the shale permeability. Also, notice that in this case there is still a flow of ions, as
the theoretical osmotic differential required for 100% membrane efficiency was not attained. As listed in Fig. 25, an ME of 9.1% was achieved. This was calculated using the following equation:

\[
ME = \frac{\Delta P}{\Pi} = \left(\frac{295}{2805}\right) \times 100 = 9.1 \%
\]

Fig. 26 shows the membrane efficiency results of native Atoka shale, i.e. \(a_w\) equal to 0.76. The result obtained indicates that the membrane efficiency was 13.2%, as calculated below:

\[
\Delta p = 374 \text{ psi} \\
\text{ME} = 13.2\%
\]
This shows that the ability of the Atoka shale to restrict the transport of ions was enhanced at this very low shale activity.

It was also observed that the bottom pressure decreased, which means that water was being driven out of the bottom chamber by the osmotic force imposed by the chemical potential difference across the shale. For a wellbore, this phenomenon would cause the hardening of the formation around the wellbore, thus improving wellbore stability and preventing pore pressure increase.

It was also observed that the permeability of the shale continued to decrease as we reduced the shale activity. The permeability of the shale at its native activity was 0.01 nD, which confirmed how constricted the pores of the shale had become at this activity. This shows the relationship between the shale permeability and its ability to maintain a stable wellbore.

<table>
<thead>
<tr>
<th>Test Number</th>
<th>Shale Aw</th>
<th>Top Fluid Aw</th>
<th>Bottom Fluid Aw</th>
<th>Permeability (nD)</th>
<th>Porosity (%)</th>
<th>Membrane Efficiency (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.98</td>
<td>0.85</td>
<td>0.98</td>
<td>0.350</td>
<td>9.56</td>
<td>0.39</td>
</tr>
<tr>
<td>2</td>
<td>0.92</td>
<td>0.85</td>
<td>0.98</td>
<td>0.214</td>
<td>7.37</td>
<td>0.79</td>
</tr>
<tr>
<td>3</td>
<td>0.86</td>
<td>0.85</td>
<td>0.98</td>
<td>0.014</td>
<td>6.75</td>
<td>9.10</td>
</tr>
<tr>
<td>4</td>
<td>0.76</td>
<td>0.85</td>
<td>0.98</td>
<td>0.010</td>
<td>5.90</td>
<td>13.20</td>
</tr>
</tbody>
</table>
Observations and Discussions

The following plots show the relationship between the membrane efficiency and the petrophysical properties of the Atoka shale at different shale activities during interaction with NaCl solutions. These values were calculated from the membrane efficiency test, the results of which are discussed above.

![Plot of membrane efficiency vs. porosity for Atoka shale](image)

**Fig. 27**: Plot of membrane efficiency vs. porosity for Atoka shale

From the plot above, we see that the membrane efficiency is negatively correlated with the Atoka shale porosity. At a porosity of about 6 %, the membrane efficiency is 13.2% and reduces to about 0.7% at a porosity of about 7.4 %. However, a further increase in porosity to about 9.6 % did not yield any significant change in membrane efficiency. This shows how the transport of ions occurs through the shale. As the pores are enlarged from...
a porosity of about 6% to 7.4%, the membrane efficiency changes with about the same gradient, but the change in membrane efficiency is very minute beyond this porosity. This suggests that after a particular porosity value is reached, further opening of the pore spaces is no longer enhancing the rate of ion transport across the shale. Hence, the membrane efficiency does not significantly change with further increase in shale porosity. This shows that mud systems that rely on osmotic backflow of formation fluid in order to control shale instability might not work effectively if the solute in the mud is NaCl and the shale porosity is more than about 7%. This would apply to NaCl muds; however, salt solutions with larger hydrated ions might have a different threshold porosity value.

![Fig. 28: Plot of membrane efficiency vs. permeability for Atoka shale](image)

Fig. 28: Plot of membrane efficiency vs. permeability for Atoka shale

Fig. 28 presents the correlation of Atoka shale membrane efficiency and permeability. For permeabilities above 0.2 nD, the shale semi-permeable membrane is virtually zero.
Figure 29 shows the positive correlation between porosity and permeability.

**Fig. 29**: Plot of permeability vs. porosity for Atoka shale

**Fig. 30**: Plot of permeability vs. shale activity for Atoka shale
Fig. 30 displays the correlation between permeability and shale water activity.

![Graph showing correlation between permeability and shale water activity](image)

**Fig. 31: Plot of membrane efficiency vs. shale activity for Atoka shale**

Fig. 31 shows the relationship between the shale activity and membrane efficiency. Given this relationship with membrane efficiency, the shale can be characterized by its native activity. The ability of shales to act as semi-permeable membranes can be deduced from their in-situ water activity. Therefore, when NaCl muds are used, low activity shales will tend to have higher membrane efficiencies and are, therefore, less susceptible to wellbore failure.
**C-5 Shale Test Results**

The membrane efficiency tests were repeated using the C-5 shale. This shale is much softer than the Atoka shale and has significantly different properties. The native porosity is 28% and it has higher clay content than the Atoka shale. Therefore C-5 has a higher swelling tendency than the Atoka shale and would thus be more troublesome when encountered during drilling operations. The porosity and permeability during its interaction with NaCl solutions will be important in describing its membrane behavior. The test results for C-5 shale hydrated to an activity of 0.86, 0.92, and 0.98, using the controlled humidity desiccators, are shown in Figs. 32, 33 and 34.
**Fig. 32:** Test 5 for C-5 shale activity = 0.86

![Graph showing pressure vs. time for Test 5 with top NaCl 0.85, bottom NaCl 0.98, \(\phi = 21.3\%\), \(K = 0.236\) nD, \(\Delta P = 3.2\) psi, 
\(ME = 0.11\%\).](image)

**Fig. 33:** Test 6 for C-5 shale activity = 0.92

![Graph showing pressure vs. time for Test 6 with top NaCl 0.85, bottom NaCl 0.98, \(\phi = 21.3\%\), \(K = 0.236\) nD, \(\Delta P = 3.2\) psi, 
\(ME = 0.11\%\).](image)
Fig. 34: Test 7 for C-5 shale activity = 0.98

Table 12 contains the porosity, permeability, and membrane efficiency for the C-5 shale as a function of water activity.

Table 12: Summary of NaCl solution in contact with C-5 shale of different activities

<table>
<thead>
<tr>
<th>Test Number</th>
<th>Shale Aw</th>
<th>Top Fluid Aw</th>
<th>Bottom Fluid Aw</th>
<th>K (nD)</th>
<th>φ</th>
<th>ME (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>0.86</td>
<td>0.85</td>
<td>0.98</td>
<td>0.012</td>
<td>16.6</td>
<td>0.37</td>
</tr>
<tr>
<td>6</td>
<td>0.92</td>
<td>0.85</td>
<td>0.98</td>
<td>0.236</td>
<td>21.3</td>
<td>0.11</td>
</tr>
<tr>
<td>7</td>
<td>0.98</td>
<td>0.85</td>
<td>0.98</td>
<td>2.513</td>
<td>23.6</td>
<td>0.03</td>
</tr>
</tbody>
</table>
It is observed that the membrane efficiency of 0.85 activity brine is generally low for this shale. It is shown that this is due to the high porosity of C-5, which is about 28% in its native state. The porosity slightly reduces to about 16.7% when the shale activity is reduced to 0.86. This is still a relatively high shale porosity, which indicates high clay content, as shown in Table 3. This will be the principal reason for the generally low membrane efficiency of C-5.

Also, observe the short time it takes the shale to achieve a near zero membrane efficiency, as well as its unsaturated state. This is indicated in the difference in the permeability of C-5 at these different activities. The effect of the permeability on the membrane efficiency has been highlighted in Atoka shale tests. Permeability was again shown to be relevant in reducing the ionic transport across the shale membrane. This ionic transport eliminates or reduces the osmotic force that is intended to draw water out of the shale and strengthen it.
**Test Series 2, with Select Water-Based Muds**

**Test Overview**

The objective of these tests was to evaluate the impact of the interaction of Atoka shale with water-based mud over time, and to study the effect of shale activity. The plan was to observe the changes that occur in shales after they contact these water-based muds, and to infer the key properties that influence the performance of these muds. For this purpose, three commercially available water-based muds were tested with the Atoka shale. Two series of tests were run for each mud, the first at an Atoka shale activity of 0.92, and the second at 0.98. Each Atoka shale sample was first tested with (a) the water-based mud, and then re-tested (b) (using the same shale sample) to reveal the changes that may have occurred in the shales’ petrophysical properties as a result of its interaction with the water-based mud during the initial testing period.

By re-testing the same shale sample with the same mud, it was hoped that the effect of exposure time could be observed. Several studies of shale interaction with water-based muds have been done using the pressure transmission test; however, these tests were usually run on the scale of hours, or a few days at best. Drilling mud exposure in the wellbore occurs for several days, giving rise to time-dependent wellbore failure. Because the flux of water and ions into and out of the shale is time-dependent, wellbore failure is also time-dependent. This testing procedure will hopefully show whether these muds can achieve wellbore stability over time, and the mechanism involved in doing so.
**Test Procedure**

Some Atoka shale samples were hydrated to an activity of 0.92, while others were hydrated to an activity of 0.98. The objective was to observe the performance of the muds at various shale activities. The shale samples were each tested twice, first (a) with a water-based mud and again, (b) after restoring the shale to its original activity using controlled humidity desiccators. Restoring the sample to its previous activity provided a direct comparison between the tests. After the original activity was re-established, the test was re-run to see whether there were any changes in the response of the shale caused by the first interaction of these muds with the shale. The procedure for this membrane efficiency experiment with water-based muds is outlined below:

- Weigh the shale sample after stabilizing it in a desiccator of known water activity
- Put the sample in the test cell and run membrane efficiency test (a)
- After equilibrium pressures are reached, calculate the membrane efficiency and permeability of the shale to ('efficiency to’ does not make sense to me, is this engineering terminology?) the mud
- Remove the shale sample from the test cell
- Re-establish its activity by placing it in the original desiccator
- Measure the weight of the mud-contaminated sample and calculate the change in porosity as \( \frac{(W_{out} - W_{in})}{\text{Volume of sample}} \)
- Re-run the membrane efficiency test (b) with the mud-altered shale
- Calculate the permeability, membrane efficiency, and the porosity a second time.
The above test, which is the first, presents the response of the 0.92 activity Atoka shale to mud A, the properties of which were listed in Chapter 3. The membrane efficiency recorded was 6.9% and the permeability to this mud was 0.030 nD. There was a significant increase in membrane efficiency, as compared to the previously tested NaCl solution. This can be attributed to an enhancement of membrane efficiency due to the mud and the higher viscosity of the mud filtrate.
After test 8a was completed, the sample was removed from the cell, re-weighed, and then placed again in the 0.92 activity desiccator. On subsequent days, the sample was removed and weighed until the weight remained constant. At that point, it was placed back in the membrane testing cell and a follow-up membrane efficiency test was run under the same conditions as before. From the weight tests, it was learned that the shale’s porosity had increased from 7.37% to 8.45% as it was hydrated back to the 0.92 activity. The membrane efficiency test for this shale is shown in Fig. 36.

![Graph showing membrane efficiency test results](image)

**Fig. 36:** Test 8b with shale (Aw = 0.92) and Mud A as test fluid- (2nd test)
Figure 36 is the result of the re-run of test 8a. This test shows that the membrane efficiency of the shale had been reduced from 6.9% to about 2.1% by the mud filtrate invasion. Also, the permeability had increased from 0.030 to 0.062 nD.

The next series of tests were also run with the Atoka shale; however, the water activity of the new shale had been increased from its native activity of 0.76 to 0.98.

**Fig. 37:** Test 9a with shale (Aw = 0.98) and Mud A as test fluid
The same set of tests shown in Fig. 35 and 36 was repeated for a shale activity of 0.98. The observed membrane efficiency was 1.15%, which was again higher than the value observed for a similar NaCl solution, but lower than that observed for shale activity of 0.86. Also, the permeability was 0.096 nD and the bottom pressure increased at this shale activity, unlike the initial test at shale activity of 0.92.

At the end of test 9a, the sample was removed, weighed, then placed back in the 0.98 desiccator, as was done for the previous tests. It was found that, as before, the porosity had increased, this time from 9.56% to 10.08%. The test is shown in Fig. 38.

**Fig. 38**: Test 9b with shale (Aw = 0.98) and Mud A as test fluid- (2nd test)
Note that the shale permeability has increased from 0.096 to 0.26nD, and the membrane efficiency has decreased from 1.15\% to 0.465\% (same trend as was observed for shale activity = 0.92)

The test procedure followed for the next four membrane efficiency tests was identical to the previous four tests, except that Mud B, the properties of which were listed in Chapter 3, was used. Also, note that Mud B had a higher activity (0.846) as compared to Mud A (0.81) or Mud C (0.79).

The sinusoidal behavior of the top pressure was a result of mud particles attempting to plug the back pressure valve.
It was observed that the membrane efficiency was essentially the same for the re-run test (10b) as it was for the first test (10a).

**Fig. 40**: Test 10b with shale (Aw = 0.92) and Mud B (2nd test)
**Fig. 41:** Test 11a with shale (Aw = 0.98) and Mud B

**Fig. 42:** Test 11b with shale (Aw = 0.98) and Mud B - (2nd test)
As was observed at a shale activity of 0.92, only a small change in the membrane efficiency (from 5.36% to 6.12%) was observed when the test was re-run at a shale activity of 0.98.

Again, the test procedure which was followed for the next four membrane efficiency tests was identical to the previous tests, except that Mud C was used instead of Mud A. Also, note that Mud C had a slightly lower activity (0.79) as compared to Mud A (0.81). This implies that Mud C imposes a slightly higher osmotic force on the bottom side of the system than Mud A.

Fig. 43: Test 12a with shale (Aw = 0.92) and Mud C as test fluid
For tests 12a and 12b, we note a large decrease in the membrane efficiency from 6.06% to 2.3%.
**Fig. 45:** Test 13a with shale (Aw = 0.98) and Mud C as test fluid

- **Top = Mud C, 0.79**
- **Bottom = NaCl, 0.98**
- $\phi = 9.56\%$
- $K = 0.038\ nD$
- $\Delta P = 87\ psi$
- $ME = 2.1\%$

**Fig. 46:** Test 13b with shale (Aw = 0.98) and Mud C-(2nd test)

- **Top = Mud C, 0.79**
- **Bottom = NaCl, 0.98**
- $\phi = 9.66\%$
- $K = 0.81\ nD$
- $\Delta P = 14\ psi$
- $ME = 0.36\%$
Again, from Figs. 45 and 46, we see that re-running Mud C produces much lower membrane efficiency. It was observed that the shale membrane efficiency decreased from 2.1% to 0.36%, with a corresponding increase in permeability from 0.038 to 0.81 nD. This relatively high permeability was probably responsible for the lack of osmotic support from the shale after contacting this mud for about 60 hours.

The results obtained for tests 8a through 13b by using the water-based muds and Atoka shale are summarized in Table 13.

**Table 13: Summary of test results for select water-based muds in contact with Atoka shale**

<table>
<thead>
<tr>
<th>Test Number</th>
<th>Shale Aw</th>
<th>Top Fluid Aw</th>
<th>Mud Type</th>
<th>Bottom Fluid Aw</th>
<th>Permeability (nD)</th>
<th>Porosity (%)</th>
<th>Membrane Efficiency (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>8a</td>
<td>0.92</td>
<td>0.81</td>
<td>Mud A</td>
<td>0.98</td>
<td>0.030</td>
<td>7.37</td>
<td>6.900</td>
</tr>
<tr>
<td>8b</td>
<td>0.92</td>
<td>0.81</td>
<td>Mud A</td>
<td>0.98</td>
<td>0.062</td>
<td>8.45</td>
<td>2.100</td>
</tr>
<tr>
<td>9a</td>
<td>0.98</td>
<td>0.81</td>
<td>Mud A</td>
<td>0.98</td>
<td>0.096</td>
<td>9.56</td>
<td>1.150</td>
</tr>
<tr>
<td>9b</td>
<td>0.98</td>
<td>0.81</td>
<td>Mud A</td>
<td>0.98</td>
<td>0.260</td>
<td>10.08</td>
<td>0.465</td>
</tr>
<tr>
<td>10a</td>
<td>0.92</td>
<td>0.85</td>
<td>Mud B</td>
<td>0.98</td>
<td>0.018</td>
<td>7.37</td>
<td>6.200</td>
</tr>
<tr>
<td>10b</td>
<td>0.92</td>
<td>0.85</td>
<td>Mud B</td>
<td>0.98</td>
<td>0.0105</td>
<td>7.98</td>
<td>6.400</td>
</tr>
<tr>
<td>11a</td>
<td>0.98</td>
<td>0.85</td>
<td>Mud B</td>
<td>0.98</td>
<td>0.0264</td>
<td>9.56</td>
<td>5.360</td>
</tr>
<tr>
<td>11b</td>
<td>0.98</td>
<td>0.85</td>
<td>Mud B</td>
<td>0.98</td>
<td>0.0063</td>
<td>9.66</td>
<td>6.120</td>
</tr>
<tr>
<td>12a</td>
<td>0.92</td>
<td>0.79</td>
<td>Mud C</td>
<td>0.98</td>
<td>0.035</td>
<td>7.37</td>
<td>6.060</td>
</tr>
<tr>
<td>12b</td>
<td>0.92</td>
<td>0.79</td>
<td>Mud C</td>
<td>0.98</td>
<td>0.0479</td>
<td>8.39</td>
<td>2.300</td>
</tr>
<tr>
<td>13a</td>
<td>0.98</td>
<td>0.79</td>
<td>Mud C</td>
<td>0.98</td>
<td>0.038</td>
<td>9.56</td>
<td>2.100</td>
</tr>
<tr>
<td>13b</td>
<td>0.98</td>
<td>0.79</td>
<td>Mud C</td>
<td>0.98</td>
<td>0.81</td>
<td>9.66</td>
<td>0.360</td>
</tr>
</tbody>
</table>

In general, note that exposure of the muds for a longer time produced a decrease in the membrane efficiencies for Muds A and C, and a small increase for Mud B.
Observations and Discussion

The results reported in Fig. 47 show that the membrane efficiency of the muds was generally higher than that of the brine. This was probably due to the fact that the muds contain membrane efficiency-enhancing agents, presumably capable of creating a semi-permeable membrane to enhance the shale’s ion selectivity. The membrane efficiency of the re-run tests was lower than the initial tests, except in the case of Mud B where it increased slightly. This suggests that unlike the other muds, mud B was capable of forming and maintaining an internal filter cake or stabilization mechanism for the Atoka shale.

Fig. 47: Change in membrane efficiency for Atoka shale (Aw = 0.92)
Figure 48 above shows that the permeability of the shale to the muds was much lower than that observed for brine. Also, there was a permeability increase in the shale after it had contacted the muds during the initial test, except for Mud B. This correlates well with the decrease in membrane efficiency observed for the other two muds and an increase in membrane efficiency observed for mud B. It can, therefore, be concluded that membrane efficiency correlates negatively with permeability. This was also observed in the tests using brine, as discussed in Test Series 1.
The figure above shows that there was an increase in porosity after the initial tests. It was also interesting that the membrane efficiency increased slightly for the mud-altered shale tests (re-run) of Mud B (see Fig. 47), despite the marginal increase in porosity. This shows that the shale’s ability to restrict ion flow is very important in determining its membrane behavior. Mud B may have enhanced this ability by reducing the permeability of the Atoka shale.
Results for Shale $Aw = 0.98$

Results using shale activity of 0.98 showed the same relationship among the porosity, permeability, and membrane efficiency as they did with shale activity at 0.92. These plots are shown below.

![Graph showing membrane efficiency for Atoka shale (Aw = 0.98)](image)

**Fig. 50:** Change in membrane efficiency for Atoka shale (Aw = 0.98)

![Graph showing change in porosity for Atoka shale (Aw = 0.98)](image)

**Fig. 51:** Change in porosity for Atoka shale (Aw = 0.98)
Figures 50 to 52 above show the same relationship and results, even though the shale activity has been increased to 0.98. However, observe that the order of magnitude of the membrane efficiency has decreased for the muds, and also for the brine. This was probably due to the fact that, at an activity of 0.98, the mean aperture diameter of the shale is larger than at an activity of 0.92. Therefore, the ability of the shale to restrict ionic movement was reduced at this activity, regardless of the mud composition. We can thus conclude that characterization of shale by their in-situ water activity is a good method for estimating their membrane performance under osmotic backflow mechanism of shale stability.
VII. Conclusions

1. Experimental results show that the membrane efficiency of Atoka shale and C-5 shale is generally low, ranging from 0.4% to about 13%. This is in agreement with researchers who have reported similar membrane efficiency values (0 to 10%) for salt solutions and shale systems (Van Ort, 2003).

2. Correlations show that the membrane efficiency is a function of the porosity and permeability of the shale.

3. Data for Atoka shale clearly show that its membrane efficiency is negatively correlated with the shale porosity until a porosity of about 7.5%. Beyond this there is essentially no change in membrane efficiency. Other shales may have different threshold porosities.

4. A good correlation was obtained between the shale permeability and the membrane efficiency. However, beyond a permeability of 0.2 nD, osmotic effects are small and there is essentially no membrane behavior.

5. For tests conducted with water-based muds, changes in permeability correlated well with change in membrane efficiency after the shale has contacted the mud.

6. This study highlights the importance of permeability-reducing agents in a mud formulation for improving the membrane efficiency of a shale.

7. Characterization of shales by their in-situ activity is a good method for predicting their stability

8. Most shales have an average pore diameter that is much larger than the average hydrated diameter of ions. This means that they will allow ion transport that mitigates osmotic backflow in wellbore stabilization. It is, therefore, important that physico-chemical permeability reduction techniques be employed in mud design for enhancing wellbore stability.
Appendix A

Permeability Model

Below is the derivation of the permeability model used in this study. It was developed by Al-Bazali et al., 2005.

\[
P_{\text{m}}
\]

\[\text{Shale Sample}\]

\[\text{Downstream Reservoir}\]

The above figure describes the experimental set up. A mathematical model is presented below for determining the shale’s permeability. In the experiments, a fluid with constant pressure \( P_{\text{m}} \) is circulating on top of the shale sample, while a reservoir under the shale is initially maintained at a pressure \( P_{\text{o}} \). It is assumed that both the shale and the downstream reservoir have an initial pressure \( P_{\text{o}} \).

The diffusivity equation, in low-permeability rocks, is used to describe the system. The partial differential equation is written as:

\[
\frac{\partial^2 P}{\partial x^2} = \frac{1}{K} \frac{\partial P}{\partial t}
\]

\( K = \frac{k}{\phi \mu c} \)
where:

- \( K \) is the shale’s diffusivity
- \( k \) is the shale’s permeability
- \( \phi \) is the porosity of the shale
- \( \mu \) is the fluid viscosity
- \( C \) is the fluid compressibility

Equation (1) describes our experimental procedure with the flowing initial and boundary conditions.

**Initial Condition:**

\[ P(x, 0) = P_o \] (The initial pressure in the shale and downstream reservoir is \( P_o \))

**Boundary Conditions:**

\[ P(0, t) = P_m \]

\[ \frac{\partial P}{\partial x}(L, t) = \frac{q\mu}{KA} \]

The system is not equipped to measure the flow rate at \( x = L \). Therefore, a modification to this boundary condition is introduced using the concept of compressibility, as follows.

\[ C = -(1/V)\frac{\partial V}{\partial P} \]

\[ \frac{\partial V}{\partial t} = -CV\frac{\partial P}{\partial t} \]

\[ q = \frac{\partial V}{\partial t} \]

\[ q = -CV\frac{\partial P}{\partial t} \]

Therefore;

\[ \frac{\partial P}{\partial x}(L, t) = -(CV\mu/KA)\frac{\partial P}{\partial t} \]

where \( \frac{\partial P}{\partial t} \) is the pressure change with time in the downstream reservoir.

Equation (1) is solved using the given boundary and initial conditions. The method of Laplace Transform is used.
\[
\frac{\partial^2 P}{\partial x^2} = \frac{1}{K} \frac{\partial P}{\partial t}
\]
\[
sP - P(x,0) = K\frac{\partial^2 P}{\partial x^2}
\]
\[
sP - P_0 = K\frac{\partial^2 P}{\partial x^2}
\]
\[
K\frac{\partial^2 P}{\partial x^2} - sP = -P_0
\]

\[
\frac{\partial^2 P}{\partial x^2} - sP / K = -P_0 / K 
\]

Equation (2) is a second order non-homogenous equation. The method of variation of parameter is used to solve equation (2).

First, a solution for the homogenous part is obtained.

\[
\frac{\partial^2 P}{\partial x^2} - sP / K = 0
\]
\[
m^2 - s / K = 0
\]
\[
m = \pm \sqrt{s / K}
\]
\[
P(x, s) = C_1 e^{\sqrt{s / K}x} + C_2 e^{-\sqrt{s / K}x}
\]

Second, a particular solution \( y_p \) for the non-homogenous part; is obtained.

Let:

\[
y_1 = e^{\sqrt{s / K}x}
\]
\[
y_2 = e^{-\sqrt{s / K}x}
\]
\[
y'_1 = \sqrt{s / K} e^{\sqrt{s / K}x}
\]
\[
y'_2 = -\sqrt{s / K} e^{-\sqrt{s / K}x}
\]

Finding the Wronskian \( W \):

\[
W = -2\sqrt{s / K}
\]
\[ y_p = -\frac{Po}{2s} - \frac{Po}{2s} \]
\[ y_p = -\frac{Po}{s} \]

Therefore, the general solution is:

\[ P(x, s) = C_1 e^{\sqrt{s/\mu}x} + C_2 e^{-\sqrt{s/\mu}x} - \frac{Po}{s} \]

\[ C_1 = \frac{A}{\beta (sP - Po)} \]

\[ C_2 = -\beta (sP - Po) \]

\[ C_1 \text{ and } C_2 \text{ are determined from the boundary conditions. The boundary condition was transformed into a Laplace form in order to solve for } C_1 \text{ and } C_2. \]

**Boundary conditions:**

\[ P(0, s) = Pm/ s \]..........................(BC1)
\[ \frac{\partial P}{\partial x(L, s)} = -(CV\mu / KA)(sP - Po) \]
\[ \text{let } \beta = CV\mu / KA \]
\[ \frac{\partial P}{\partial x(L, s)} = -\beta (sP - Po) \]..........................(BC2)

Applying the boundary conditions yields the following equations, which are solved to obtain \( C_1 \) and \( C_2 \).

**Applying BC1:**

\[ Pm/ s = C_1 + C_2 - \frac{Po}{s} \]
\[ C_1 + C_2 = 1/ s(Pm + Po) \]
\[ C_1 + C_2 = \frac{1}{s(Pm + Po)} \]..........................(4)

\[ \text{where;} \]
\[ A = \frac{1}{s(Pm + Po)} \]

**Applying BC2:**

\[ -\beta (sP - Po) = \alpha C_1 e^{\alpha x} - \alpha C_2 e^{-\alpha x} \]

\[ \text{where;} \]
\[ \alpha = \sqrt{s/\mu} \]
Solving equations (4) and (5) for $C_1$ and $C_2$.

\[
C_1 = \frac{(DA+E)}{(D-B)}
\]
\[
C_2 = \frac{(BA-E)}{(B-D)}
\]

\[
P(x,s) = \left(\frac{(DA+E)}{(D-B)}\right)e^{\frac{sE}{sK}}x + \left(\frac{(BA-E)}{(B-D)}\right)e^{-\frac{sE}{sK}}x - Po/s \quad \ldots \ldots (6)
\]

From the literature, a solution to equation (6) was obtained. As given in Carslaw and Jaeger’s book, titled “Conduction of Heat in Solids,” and Van Oort et al (1994), a solution to equation (1) that satisfies the boundary and initial conditions is given below.

The solution can be expressed as:

\[
(P(x,t) - Po)/(Pm - Po) = 1 - 2 \sum_{n=1}^{\infty} EXP(-\phi_n^2 Kt/L^2) \sin(x\phi_n/L)/(\cos \phi_n \sin \phi_n + \phi_n) \quad \ldots \ldots (7)
\]

where;

\[
\phi_n \tan \phi_n = AL\phi/V
\]

\[
\phi \quad \text{is the porosity of the shale sample}
\]
\[
V \quad \text{is the downstream reservoir volume}
\]
\[
L \quad \text{is the length of the shale sample}
\]
\[
A \quad \text{is the cross-sectional area of the shale sample}
\]

Van Oort et al (1994) states that the parameter $\phi_n$ depends strongly on the ratio of the sample pore volume to the downstream reservoir volume. He further explains that when
this ratio is small, as is the case for our experimental system, equation (7) reduces to the following equation at \( x = L \).

\[
\frac{(P(L,t) - P_o)}{(P_m - P_o)} = 1 - \exp \left( -\frac{Akt}{\mu CVL} \right)
\]

where \( P(L,t) \) is the pressure of the downstream reservoir, which equals the pressure of the shale sample at \( x = L \).

Taking the natural logarithm of equation (7) yields:

\[
\ln \left( \frac{(P_m - P(L,t))}{(P_m - P_o)} \right) = \lambda t
\]

where \( \lambda = -\frac{Ak}{\mu CVL} \)

\[
\lambda = \ln \left( \frac{(P_m - P(L,t))}{(P_m - P_o)} \right) / t
\]

According to equation (8), the plot of \( \ln \left( \frac{(P_m - P(L,t))}{(P_m - P_o)} \right) \) versus time provides the slope of the line equals \( \lambda \).

The following procedure is adopted in order to estimate the permeability of the shale sample.

- Pressurize the downstream reservoir up to the desired value.
- Allow the downstream pressure to stabilize and let this pressure be \( P_o \). The downstream pressure should equal the shale’s pore pressure.
- Start flowing upstream fluid on top of the shale at \( P_m \).
- Record the downstream pressure with time; this is \( P(L,t) \).
- Plot \( \ln \left( \frac{(P_m - P(L,t))}{(P_m - P_o)} \right) \) versus time.
- Determine the slope of the line (\( \lambda \)).
- Calculate the shale permeability, as follows:

\[
k = \left( \frac{\lambda \mu CVL}{-A} \right)
\]
Appendix B

Leak Tests

In order to determine the membrane efficiency of the samples, it was necessary that the bottom chamber be completely sealed off. In order to evaluate this, a solid brass disk was inserted into the test cell, in place of the shale sample. The lower chamber was pressurized to about 400 psi and valve V2 was closed to isolate the 1.3 ml bottom volume. Present measurements were taken over time.

Figure B1 shows that the chamber was initially losing pressure at about 47 psi/hr. However, by loosening and tightening the bottom pressure transducer, the leak was reduced to 0.704 psi/hr, as shown in Fig. B2. Finally, after replacing the o-rings with new ones, the leak was reduced to 0.06 psi/hr (Fig. B3).

![Bottom System Leak Test (6/14/2007)](image)

**Fig. B1:** System leak test 1
Fig. B2: System leak test 2

Fig. B3: System leak test 3
Appendix C

Equipment calibration

**Fig. C1:** Hand Pump Calibration.

Because water was introduced into the bottom chamber using the hand pump, during certain test, it was necessary to calibrate the hand pump to ensure its accuracy. The result is shown in Fig C1; the initial noise in the data is probably due to air compressibility effects.

The zero-volume transducer used in the bottom chamber was calibrated using a load cell and was found to have an error between 0.8 and 1.6%
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