MAKING SUCCESSFUL PERMEABILITY MEASUREMENTS WITH ASPHALTIC CRUDE OILS

Kory Holmes[1], Gary Potter[1], Dan Scott[1], Wade Williams[1], Tom Fate[2]
[1]Core Laboratories, Houston, Texas
[2]ChevronTexaco, Houston, Texas

ABSTRACT

Reservoirs with asphaltic oils are notorious for being difficult to evaluate with respect to reservoir properties. This is largely due to the flocculation of dispersed asphaltenes when the rock and fluids are brought to the surface. The flocculated asphaltenes deposit on the rock surface affecting the wettability and are suspended in the oil causing problems with flow measurements. Cleaning and aging procedures attempt to restore the rock surface back to reservoir conditions. However, unless all asphaltenes are removed, flocculated material will continue to form. Complete removal of the asphaltenes would alter the chemical and physical characteristics of the oil, creating questions about the applicability of the flow tests.

This paper provides successful results from relative permeability tests using asphaltic oils. Attempts to measure oil permeabilities with dead crude oil have resulted in a continually decreasing permeability. However, once the crude oil was recombined to live oil, stable oil permeability was measured. The relative permeability tests with live oil were successfully run without the sample plugging problems typical of dead oil with flocculated asphaltenes. Results are shown for both sandstone and carbonate reservoirs.

INTRODUCTION

The complexities of heavy oil asphaltene molecules, and associated asphaltene flocculation, make production flow management a complex and expensive problem that must be solved in virtually every oil-producing region around the world.

Asphaltene flocculation can cause problems in laboratory tests in two ways: pore throat plugging and wettability alteration. With some crude oils, small asphaltene aggregates pass through the pore system and do not plug or bridge pore spaces. In other cases, as little as 1-5 wt% asphaltenes at or near onset of flocculation, can hinder successful relative permeability testing by substantially lowering effective oil permeability at initial water saturation and K_o/K_{eff} values.

This paper presents three methods used in the laboratory to deal with asphaltene flocculation and perform tests properly in order to produce useable data. One of these methods is usually chosen based on client needs, which are dictated by quantity of live or dead crude oil available, project timing restrictions, and project funding resources.

STATEMENT OF THE PROBLEM

How can flow properties of asphaltic reservoirs be reliably measured? These flow properties include effective oil permeability at initial water saturation (Swi) and relative permeability.

Since flocculating-asphaltenes clog the sample, the most direct approach might appear to be removal of the asphaltenes, by filtration or centrifugation, before they can deposit in the sample. However, attempts to remove the asphaltenes by centrifuge are generally not successful because of the small density difference between the dispersed asphaltenes and the rest of the crude oil. Removing flocculated asphaltenes by filtration can also be unsuccessful as illustrated by the continued clogging by asphaltic oils after having been filtered with 0.5 and smaller micron filters before loading into the flow apparatus. Therefore, it is best to remove the flocculated asphaltenes just before injection into the test sample. Our first example is of a method in which the crude oil is injected through a sacrificial core, installed immediately upstream of the core sample.

Since flocculation can occur with a reduction of crude oil pressure, one strategy is to obtain samples and run all tests at pore pressures above the flocculation pressure. However, it is difficult and expensive to collect enough pressurized crude oil and keep the oil at pressure for completion of a test. Returning the dead crude oil to high pressure can keep additional asphaltenes from flocculating. However, measurements have shown that a considerably higher pressure must be achieved than the flocculation pressure to re-disperse flocculated asphaltenes. As an example, a crude oil that had an asphaltenes flocculation point measured at 5300 psi, required pore pressures in excess of 7000 psi before stable oil permeability could be measured. Running flow tests with such a high pore pressure presents experimental challenges when high net overburden pressure is also required.

To help reduce the operational challenges with running very high system pressure, we explored the use of live crude oils at lower pressure. For two fields, we found that stable crude oil permeabilities could be measured once live crude oil was used.

If live oil cannot be used for the flow tests, the samples can be restored with dead crude at high pore pressure and then the crude oil can be replaced with refined mineral oil.

This paper discusses our experience using these approaches to solving sample clogging due to flocculated asphaltenes.

REMOVING FLOCCULATED ASPHALTENES

Even though before every test the crude oil is pre-filtered, clogging of the sample still continues to occur. Removing flocculated asphaltenes by pre-filtering, often days before injection into the test sample, allows time for the dispersed asphaltenes to re-flocculate. The following example shows success in using a sacrificial plug immediately inline before the test sample.

Test Procedures

The oil was spun in a centrifuge to remove mud solids and water, and was pre-filtered with a 0.45 micron Cuno filter. One-inch diameter preserved state samples were flushed with brine to remove mud solids, and Swi was established by centrifuge. The samples were briefly vacuum saturated with depolarized kerosene. Kerosene is depolarized by flushing through a column of activated silica to remove polar compounds. The samples were loaded into a hydrostatic core holder, and depolarized kerosene was injected to establish pore pressure before elevating the temperature to a reservoir temperature of 210°F. Permeability to depolarized kerosene in a forward direction was measured at room condition and reservoir condition. Crude oil was injected in the forward direction to displace the depolarized kerosene and immediately damage began occurring (see Figure 1). Depolarized kerosene was injected in the forward direction, and then injected in the reverse direction. A sacrificial core plug was placed in the flow system, just before the test sample. The sacrificial core plug temperature was elevated to and maintained at 160°F. Crude oil was injected through the sacrificial core and into the test sample. The sacrificial core was changed after every test, to maintain acceptable upstream pressures.

Results

The crude oil permeability was reduced from 160 mD after three pore volumes of injection, to 59 mD. An additional pore volume of crude oil injection reduced the permeability to 55 mD. Depolarized kerosene was injected, in the forward direction, to displace the crude oil and the permeability recovered to 143 mD where it stabilized. A reverse permeability to depolarized kerosene was performed and the permeability increased to 156 mD, and stabilized at that value. Recovery of lost permeability was noted after reverse direction injection of xylene increased the K_{eff} almost to the initial value [3]. Crude oil was injected through the sacrificial core and into the test sample. The differential pressure stabilized across the test sample, but the upstream pressure (due to the plugging of the sacrificial core) continued to climb throughout the crude oil injection.

USING A LIVE CRUDE OIL

Case 1

These tests began with the restoration of the samples with dead crude oil. The dead crude oil had a predicted asphaltene content, based on Pederson et al. [4], and Riazi and Daubert [5], of approximately 3 wt%. Immediately, asphaltene deposition began clogging the sample. Consequently, the number of pore volumes injected was limited. The sample was aged at reservoir temperature for 4 weeks and then live crude was injected.

Test Procedures

Dead reservoir crude oil was centrifuged to remove water and mud solids and then filtered to 0.2 microns prior to use. Two cleaned and dried core samples from a carbonate reservoir were saturated with brine and spun to initial water saturation in a high-speed centrifuge in an air-displacing-brine configuration. The samples were briefly vacuum saturated with depolarized kerosene. The samples were loaded into a hydrostatic coreholder and the appropriate net confining stress applied. The temperature of the system was elevated to reservoir conditions (250° to 280°F) while bypassing dead crude oil around the sample. The dead crude oil was injected at reservoir temperature at rates between 1.5 to 3 ml/min. The samples were allowed to age in the crude oil at 200 psi at 250°F and 280°F, respectively. At the end of the wettability restoration, the dead crude oil was displaced with live reservoir crude oil from a bottom hole sample. Several pore volumes of live oil were injected, and no plugging was noted. Permeability remained stable throughout the entire injection.

Results

Dead crude oil plugging was detected immediately, so the injection was limited to between 5-10 pore volumes of through-put prior to aging. After aging live crude oil was injected to displace the dead crude oil, and a stable live crude oil permeability was measured, albeit a lower permeability. The lower permeability was partially the result of a wettability change during aging and possibly deposition of the previously flocculated asphaltenes in the pore space. Figures 2 and 3 illustrate the permeability sequences of these samples during restoration.

Case 2

This is another example of the benefits of a live crude oil to get stable oil permeability. Other laboratories had previously attempted to measure effective oil permeability with dead crude oil. Measurement using the Institute of Petroleum-143 method (similar to SARA analysis) indicated 2.34 wt% asphaltenes present in the stock tank oil. The result was a continually decreasing permeability as the dead crude oil was injected. The dead crude oil was displaced with live recombined reservoir oil. Stable oil permeability was measured, before and after aging, using live reservoir crude oil.

Test Procedures

Samples from a Gulf of Mexico sandstone reservoir were selected for relative permeability testing after wettability restoration. Supplied dead reservoir crude oil was dewatered by centrifugation, and filtered to 0.2 microns prior to use. Stock tank oil and a synthetic gas were recombined to reservoir conditions for the water-oil relative permeability testing. Selected core plugs were miscibly cleaned with toluene, chloroform, and methanol. The methanol was displaced with brine and the samples were unloaded. Each plug was placed in a hydrostatic coreholder and porous plate desaturated using nitrogen at 200 psi. Each sample was unloaded weighed. The samples were briefly vacuum saturated in depolarized kerosene, loaded into a coreholder, and the desired net confining stress and pore pressure established. The temperature was elevated to 163° F, while bypassing depolarized kerosene around the sample. Dead crude oil was injected at rates between 1 and 2 ml/min. Injection was limited to 3-5 pore volumes of dead oil. The pore pressure was elevated to 500 psi above the bubble point, and 3-5 pore volumes of live crude oil was injected to displace the dead crude oil. No plugging was detected, and a stable permeability was achieved. The samples were allowed to age for four weeks. At the end of the wettability restoration, 5-10 pore volumes of the live oil was injected and permeability to oil at initial water saturation measured.

Results

After the temperature had stabilized, dead crude oil was injected at rates between 1 and 2 ml/min. Plugging was detected immediately, so the injection was limited to 3-5 pore volumes of dead oil. When live crude oil was injected to displace the dead crude oil, no plugging was detected, and a stable permeability was achieved. The samples were allowed to age in the live crude oil at pressure under net confining stress at 163°F for four weeks. At the end of the wettability restoration, 5-10 pore volumes of the live oil was injected and permeability to oil at initial water saturation measured. No plugging was noted, and permeability remained stable throughout the injection. Permeability versus throughput results are shown in Figures 4 and 5.

REPLACING THE CRUDE OIL WITH REFINED MINERAL OIL

In some cases, it is not possible to use live crude oil for reasons such as oil sample quality, amount of oil available, or cost. In this example, a set of samples was aged with dead crude oil at 1700 psi above the asphaltene precipitation pressure. A sacrificial core was placed, inline, upstream of the sample. After 3 weeks of aging, the dead crude oil was displaced with mineral oil. Though permeability to the dead crude oil decreased during the aging process, a portion of the permeability loss was regained and wettability was preserved with injection of refined mineral oil. Asphaltene content of this crude oil is shown in Table 1.

Test Procedures

Sandstone samples with a varying range of air permeability were selected for relative permeability testing after wettability restoration. The core plugs were miscibly cleaned with toluene, chloroform, and methanol. The samples were dried and basic properties were measured. Supplied dead reservoir crude oil was dewatered by centrifugation, and filtered to 0.45 microns prior to use. The samples were pressure saturated with synthetic formation brine, and desaturated to initial water saturation by centrifugation at 400 psi capillary pressure in an air-brine system. The samples were briefly vacuum saturated in depolarized kerosene, loaded into a hydrostatic coreholder, and 500 psi pore pressure and 4150 psi net confining stress applied. The temperature was elevated to 300° F, while bypassing depolarized kerosene around the sample. Effective kerosene permeability at Swi was determined. Pore pressure was elevated to 7000 psi while reducing net overburden to 1700 psi, due to coreholder limitations. Dead crude oil was injected through a sacrificial core at the reservoir temperature at rates between 1 and 2 ml/min. Injection was limited to 3-5 pore volumes of dead oil. The samples were allowed to age in the dead crude oil for 52 days.

Results

Dead crude oil permeability measurements indicated a dramatic reduction when compared with the depolarized kerosene permeability. We attribute this permeability reduction to wettability alteration and not asphaltene flocculation, due to the fact that the permeability stabilized at the reduced value and did not continue its downward trend.

Periodic effective crude oil permeability measurements are indicated in figure 6. At the end of the wettability restoration, 5-10 pore volumes of a mineral oil was injected and permeability to oil at initial water saturation measured. No plugging was noted and a portion of the original permeability was recovered, while preserving the altered wettability, as shown on Figure 7.

CONCLUSIONS

Removal of Flocculated Asphaltenes

The removal of flocculated asphaltenes by centrifugation, in our experience, has limited effectiveness. However, testing has shown that some asphaltic dead crude oil can be filtered out effectively if the filtration is performed just prior to injection into the core plug. Prior to using enhanced filtration, asphaltene deposition reduced effective oil permeability by 67%, with just four pore volumes of crude oil injection. Forward and reverse injection of depolarized kerosene recovered most of the lost permeability. The filtration method included the use of a sacrificial Berea plug placed just upstream of the test plug. Permeability of the sacrificial Berea plug continually decreased with throughput, however, the permeability of the test sample remained stable throughout testing.

Using a Live Crude Oil

Asphaltic dead crude oil permeability measurements have shown to be unstable in most cases, when measured below the flocculation pressure. And testing with pore pressures above the flocculation pressure has limited laboratory use due to equipment limitations and the long duration needed for wettability restoration. Using live crude oil allows for a reduction in pore pressure to a more manageable level, therefore increasing the number of successfully completed tests. Live crude oil permeability measurements have shown to be stable, and in some instances have reversed some of the permeability reduction caused by the flocculated asphaltenes in dead crude oils. Figures 2, 3, 4, and 5 indicate a continuing reduction of dead crude oil permeability with throughput. These figures also show the stabilization of permeability measurements made with live crude oil, and in, Case 2, some recovery of permeability lost to asphaltene deposition during dead crude oil injection.

Replacing the Crude Oil with Refined Mineral Oil

Dead asphaltic crude oil, at pore pressures above flocculation pressure, can be used for sample wettability restoration. The lower net confining stress, due to equipment limitations, will not affect the accuracy of the subsequent test measurements. After wettability restoration, mineral oil is injected to displace the dead crude oil, prior to reducing the temperature and pore pressure. We have used this method when costs of acquiring or shipping live crude oil have made this alternative unavailable. Figure 6 indicates that the dead crude oil permeability measurements were stable after the initial permeability reduction due to wettability changes during restoration. Injecting mineral oil to displace the dead crude oil recovered a small percentage of the permeability lost during dead crude oil injection and the wettability restoration period. Figure 7 illustrates that the character of the relative permeability curves still exhibit properties, typical of oil-wet samples.

ACKNOWLEDGEMENTS

Support for this work was provided by ChevronTexaco (USA) and Core Laboratories-Advanced Technology Center (Houston). We would also like to acknowledge Kyle Bradford, for his research contribution to this project.

REFERENCES

1. Escobedo, J. and Mansoori, G.A. "Viscometric Principles of Onsets of Colloidal Asphaltene Flocculation in Paraffinic Oils and Asphaltene Micellization in Aromatics", SPE Production & Facilities, (1997), May, 116.
2. Leontaritis, K.J. "Asphaltene Deposition: A Comprehensive Description of Problem Manifestations and Modeling Approaches", SPE 18892, March, 1989.
3. Turta, A.T., and Najman, J. and Singhal, A.K., and Leggit, Shelly and Fisher, Douglas, "Permeability Impairment due to Asphaltene During Gas Miscible Flooding and its Mitigation", SPE 37287, February 1997.
4. Pederson, K. S., Thomassen, P., and Fresenslund, Aa., "Thermodynamics of Petroleum Mixtures Containing Heavy Hydrocarbons. 1. Phase Envelope Calculations by Use of the Soave-Redlich-Kwong Equation of State," *Ind. Eng. Chem. Process Des. Dev.*, 23, 1984, pp 163-170.
5. Riazi, M. R. and Daubert, T. E., "Prediction of the Composition of Petroleum Fractions," *Ind. Eng. Chem. Process Des. Dev., 19, 1980b, pp. 289-294.*

Table 1. Asphaltene Content of Stock Tank Oils

STOCK TANK OIL ASPHALTENE CONTENT

Sample ID	De-Asphalting Treatment	Asphaltene Content, wt%
Crude Oil 1		
Original Sample	None	2.53
Centrifuged	Centrifuged 2 hours at 1500 rpm & 150°F	2.58
Centrifuged	Centrifuged 16 hours at 1500 rpm & 150°F	2.65
Crude Oil 2		
Original Sample	None	6.22
Centrifuged	Centrifuged 4 hours at 1500 rpm & 150°F	5.67
Centrifuged	Centrifuged 16 hours at 1700 rpm & 150°F	6.61
Centrifuged-Rerun	Centrifuged 16 hours at 1700 rpm & 150°F	6.49

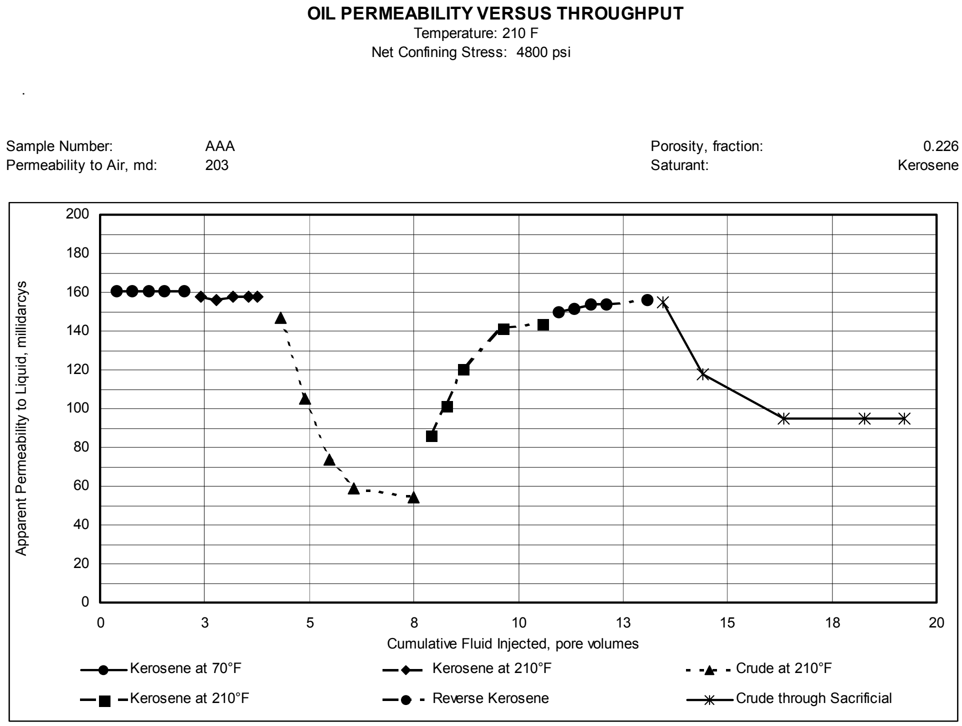

Figure 1. Oil Permeability Versus Throughput with Sacrificial Core.

OIL PERMEABILITY VERSUS THROUGHPUT
Temperature: 280 F
Net Confining Stress: 2400 psi

Sample Number:	H19	Porosity, fraction:	0.182
Permeability to Air, md:	42.8	Saturant:	Dead Crude Oil

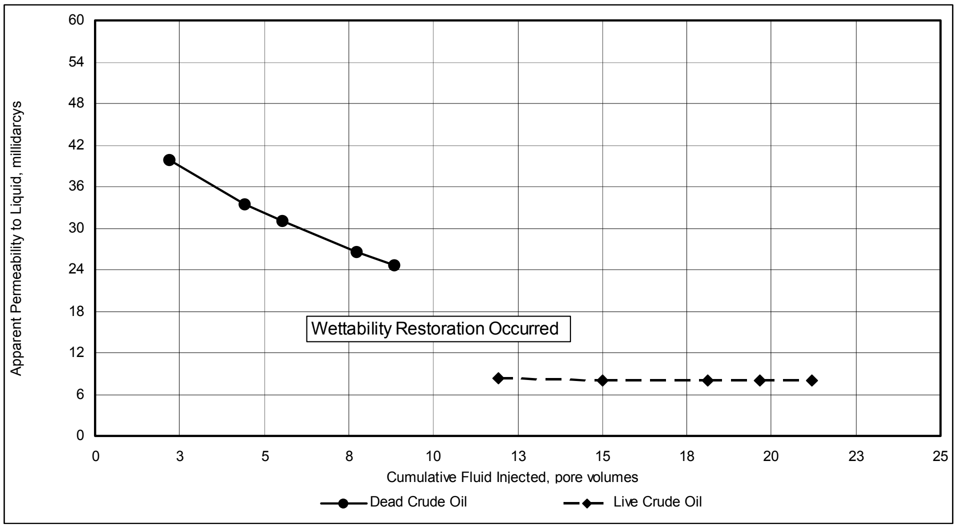

Figure 2. Oil Permeability Versus Throughput (Live Oil Case 1).

OIL PERMEABILITY VERSUS THROUGHPUT
Temperature: 250 F
Net Confining Stress: 2400 psi

Sample Number:	H5	Porosity, fraction:	0.128
Permeability to Air, md:	20.6	Saturant:	Dead Crude Oil

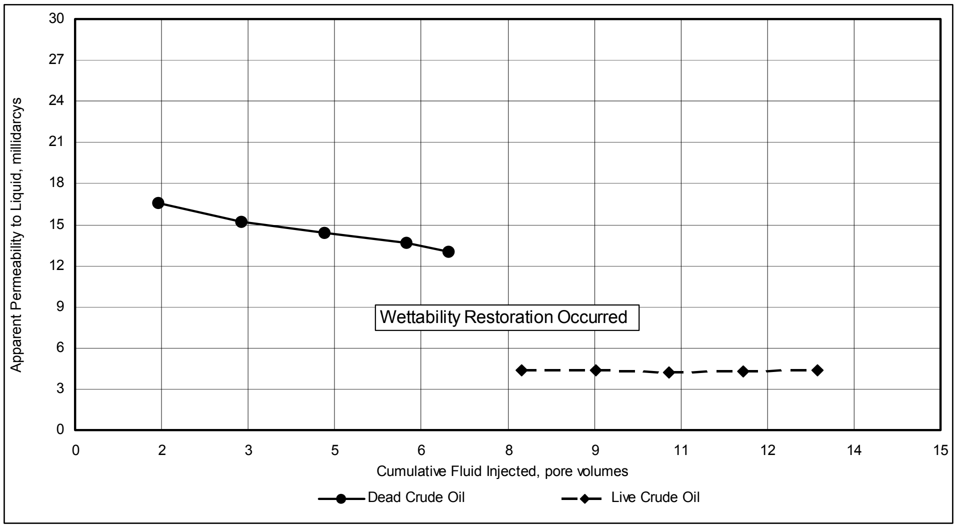

Figure 3. Oil Permeability Versus Throughput (Live Oil Case 1).

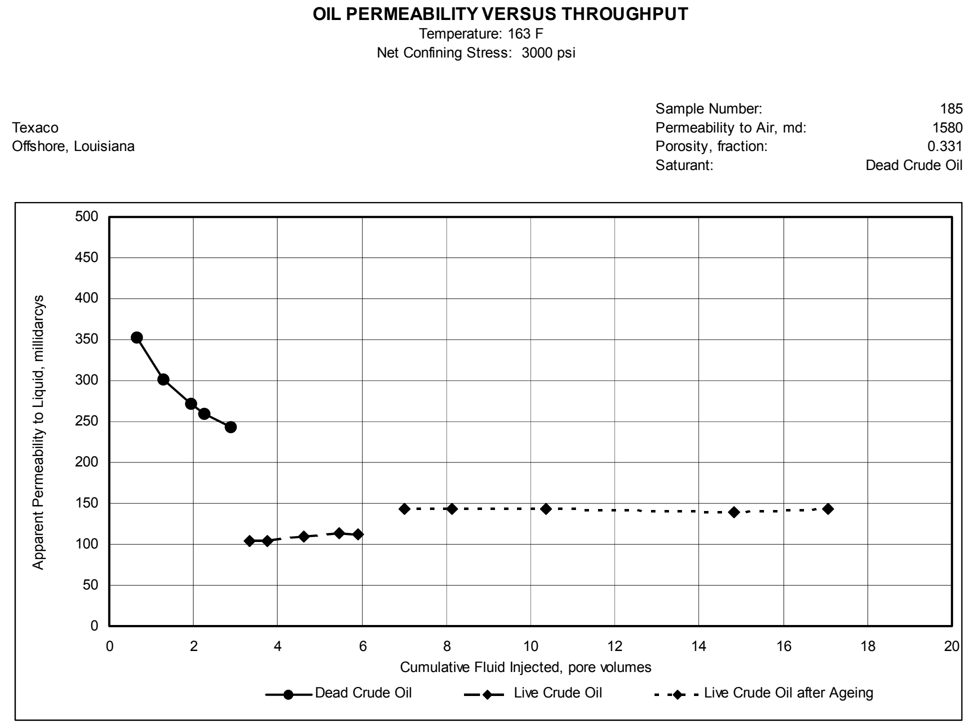

Figure 4. Oil Permeability Versus Throughput (Live Oil Case 2).

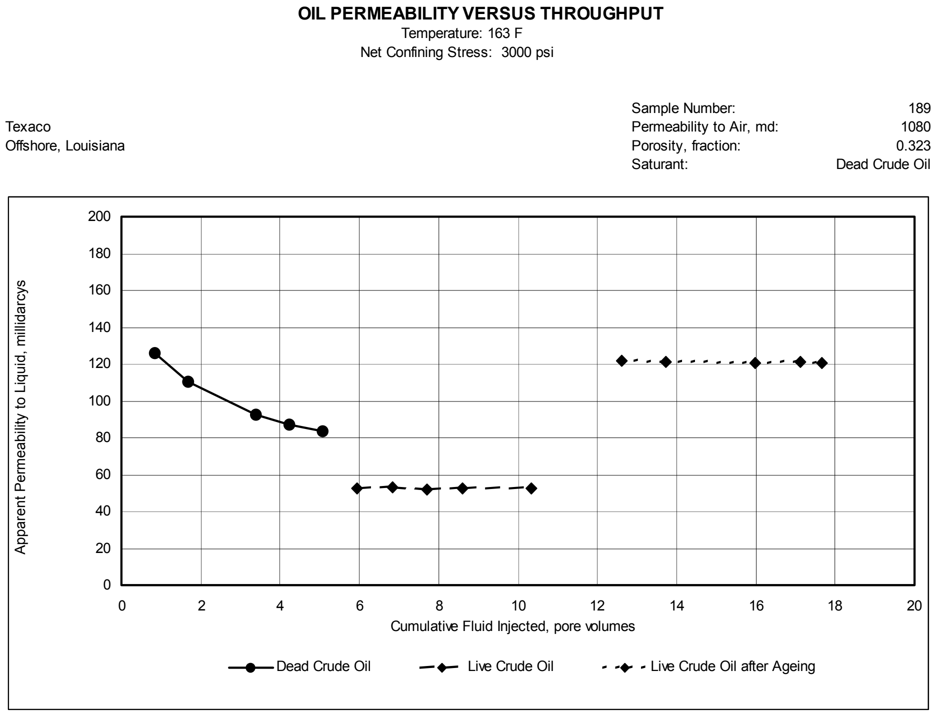

Figure 5. Oil Permeability Versus Throughput (Live Oil Case 2).

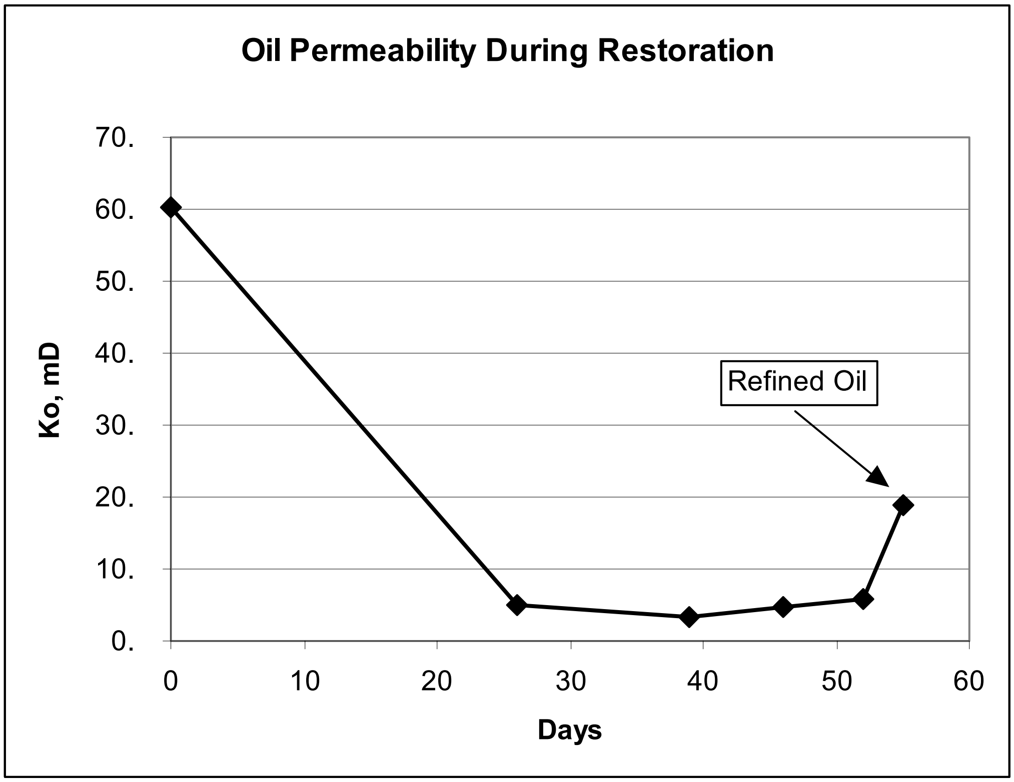

Figure 6. Oil Permeability Versus Time (Aging with Crude, and Final Permeability to Mineral Oil).

WATER - OIL RELATIVE PERMEABILITY

Steady-State Method Restored-State Sample
Net Confining Stress: 4644
Temperature: Ambient

Sample Number:	B1
Sample Depth, ft:	13830.1
Permeability to Air, mD:	76.3
Porosity, fraction:	0.132
Initial Water Saturation, fraction:	0.160
Effective Oil Permeability at Swi, mD:	65.5

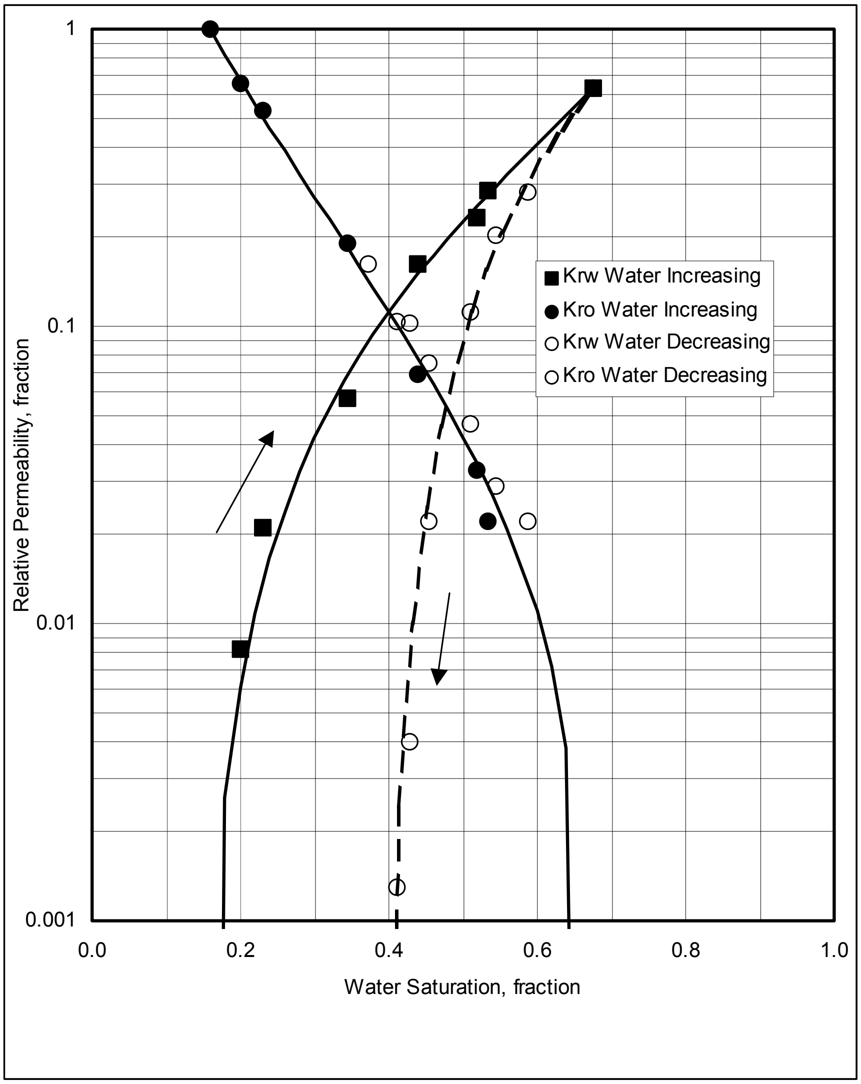

Figure 7. Water-Oil Relative Permeability with Mineral Oil (After Crude Oil Wettability Restoration).