NEW PROCEDURE AND APPARATUS FOR COMBINED RESERVOIR CONDITION GAS-BRINE RELATIVE PERMEABILITY, CAPILLARY PRESSURE, AND ELECTRICAL PROPERTY MEASUREMENTS

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ABSTRACT

In designing special core analysis programs for the deep Norphlet sandstone found in the U.S. Gulf of Mexico offshore Alabama, we found that several experimental conditions warranted a new approach. Simulating reservoir conditions required net confining pressures of 55 MPa, temperatures of 150°C, and a brine salinity exceeding 200,000 ppm. Program objectives included achieving brine saturations in the range found in situ before conducting imbibition and drainage steady-state relative permeability and resistivity measurements.

The test sequence required driving a fully brine-saturated sample to a connate brine saturation condition as a starting point for the imbibition portion of the relative permeability experiment. Desaturation with gas flow did not adequately drive plugs to irreducible brine saturation; therefore, capillary pressure desaturation was necessary to complete this process. A method was devised to desaturate a core plug through a porous plate, then alternatively bypass that flow path and conduct normal steady-state two-phase flow experiments. A new endpiece device for the outflow end of the test apparatus was designed and patented to allow this procedure. The endpiece provides capabilities to desaturate to a desired saturation range and to continue with flow, resistivity, or capillary pressure versus saturation measurements without having to disturb the sample or cycle between ambient and test temperature and pressure conditions.

Plug permeabilities and saturations were significantly affected by the moisture content of the gas. Humidifying the gas so that it neither added nor subtracted water from brine within core plugs proved to be challenging. Plug saturations were determined from X-ray scans and resistivity measurements. The effects of under- and oversaturating gas with water vapor were identified from significant changes in resistivity versus X-ray-determined saturation trends at low brine saturations. Once identified, these effects were eliminated by fine-tuning the humidifier.

The new procedures and designs developed for this project yielded data sets which were adequate for accomplishing the goals of the core analysis program.

SYSTEM DESCRIPTION AND DESIGN CONSIDERATIONS

Figure 1 is a flow system schematic of the steady-state gas-brine relative permeability apparatus that was used for this project. For simplicity, some of the hardware (such as pressure relief valves and X-ray equipment) is not shown. Nitrogen was used as the gas phase in these experiments. Gas injection rates were adjusted by two mass flow controllers with 0 to 10 and 0 to 2,000 standard cubic centimeter per minute capacities. Gas was heated and humidified upstream from the core plug.

Brine injection rates were controlled by a high-pressure syringe pump. Brine at the test pressure was preheated upstream from the plug. The brine and coreholder were heated in a hot-air oven.

Plugs were tested under simulated reservoir net confining pressure and temperature conditions within a coreholder. Sandstone inserts in the plug endpieces mixed fluids and reduced capillary end effects. Pressures were measured at the test-sample faces. Pressure drop and electrical resistance were measured from the sample inlet to outlet faces. The endpiece at the outlet end of the plug contained a porous ceramic disk in addition to the sandstone segment. This design allowed us to desaturate to a desired level and to continue with flow, resistivity, or capillary pressure versus saturation measurements without having to disturb the sample. A patent was granted for the design and application of this unique endpiece device.[1]

Backpressure was maintained on the system. Downstream from the core plug, gas and brine were separated, and the gas was cooled to the laboratory temperature before it left the system through the backpressure regulator. Limiting flow through the backpressure regulator to single-phase gas flow provided more stable pressures than those that would have resulted with two-phase flow through the regulator. Flowing only gas through the regulator also minimized corrosion and plugging within the backpressure regulator.

Rock fluid saturations were determined from X-ray absorption and from resistivity measurement techniques. Saturations calculated by each method were checked against each other.

The following paragraphs describe key components of the system.

Coreholder: The coreholder was designed for confining pressure and temperature to 55 MPa and 150°C. The design used few O-ring seals to reduce the risk of high-temperature seal failure. The 0.5 cm thick walls of the titanium coreholder were sufficient to meet test temperature and pressure constraints while allowing the use of X-ray absorption methods to measure core fluid saturations. The coreholder had one fixed and one floating endpiece. Plug endpieces were electrically isolated for resistivity measurements.

End-pieces: Porous disk desaturation was specified to drive brine saturations to about 20% at the start of each test. Initial plans were to desaturate with an endpiece containing a porous disk, then switch to a flow endpiece. Changing endpieces during a test was impractical for several reasons. A unique endpiece was designed for both flow and desaturation steps. Figure 2 is a schematic of the downstream endpiece.

The plug endpieces contain fired Berea sandstone inserts. These inserts mix fluids upstream from the plug and reduce capillary end effects at the upstream and downstream plug faces. Both endpieces have ports for pressure measurements at the plug faces. Pressures, pressure drops, and electrical resistance are measured from inlet to outlet plug faces. The outlet endpiece contains a porous ceramic disk in addition to the Berea sandstone segment. The outlet endpiece provides capabilities to desaturate to a desired saturation range and to continue with flow, resistivity, or capillary pressure versus saturation measurements without having to disturb the sample or cycle pressure conditions. Changing from porous disk desaturation to normal flow production is accomplished by simply switching a three-way valve as shown schematically in figure 1.

Temperature control: Gas was heated in a humidifier. The humidifier temperature was maintained by a temperature controller and heating tape. Flowlines were well insulated between the humidifier and coreholder.

A hot-air oven with forced circulation was built to heat the coreholder and to preheat brine. Core plugs within coreholders were X-ray scanned through the oven.

Gas Humidifier: Gas was saturated with water vapor inside a temperature-controlled cylinder. The cylinder was partially filled with water. A sight tube connected from the bottom to the top of the cylinder was used to determine when to refill the humidifier. Gas was injected through a 2-μm sintered stainless-steel filter element into the bottom of the cylinder. The gas was saturated with water vapor as it passed through the water column and during its residence time within the humidifier. Pure water was used as the saturating fluid. The humidifier temperature was set so that the vapor pressure of water vapor in the humidifier matched the vapor pressure of water vapor over brine within the core plug at the average coreholder pressure and temperature conditions.

Viscosity measurements and calculations: Brine viscosities were measured in a capillary tube viscometer at the 150°C test temperature. The viscometer was calibrated against Cannon-Fenske results and by comparing physical measurements for several fluids at various temperatures with data from the literature. Nitrogen was used as the test gas. The humidified nitrogen viscosity was calculated using the following equation:[2]

$$\mu_{mixture} = \frac{\sum_i \left(\mu_i * \chi_i * \sqrt{MW_i}\right)}{\sum_i \left(\chi_i * \sqrt{MW_i}\right)} \quad (1)$$

where

μ_i = viscosity of gas i, cP

MW_i = molecular weight of gas i, g/mol

χ_i = mole fraction of gas i, fraction

With nitrogen and water vapor as the components of the gas mixture, equation 1 becomes

$$\mu_{saturated\ N_2} = \frac{\left(\mu_{dry\ N_2} * \chi_{dry\ N_2} * \sqrt{MW_{N_2}} + \mu_{water\ vapor} * \chi_{water\ vapor} * \sqrt{MW_{H_2O}}\right)}{\left(\chi_{dry\ N_2} * \sqrt{MW_{N_2}} + \chi_{water\ vapor} * \sqrt{MW_{H_2O}}\right)} \quad (2)$$

Saturation Measurements: Saturations were measured within plug samples using a linear X-ray scanning device. X-ray scans of plugs were taken through the oven and coreholder. The X-ray beam was collimated to provide a spot diameter of about 10 mm at the core plug location.

Plugs were X-ray scanned lengthwise in 5 mm increments. Scanner movement and data acquisition were controlled by a laboratory computer. X-ray calibrations were determined for each scan position within each plug so that saturations could be determined from X-ray results. The test brine was doped with sodium iodide (6.5% to 7.5% by weight depending upon other test parameters) to increase contrast between gas and brine X-ray absorption characteristics. Figure 3 shows typical X-ray spectra data for one scan position within a rock plug when the plug was saturated with brine. The Y-axis is intensity in photon counts per second while the X-axis is photon energy in keV. Intensities were integrated over an energy range of interest, such as from 40 to 60 keV on figure 3, yielding a single intensity number for saturation calculations. The natural log of intensity varied linearly with brine saturation. Calibrations to determine brine saturations from X-ray data were constructed for each scan position using dry and brine saturated X-ray scan data. Saturation results for all scan positions were averaged to represent rock conditions for relative permeability and resistivity functions. These X-ray techniques are

similar to those described in a paper by Potter and Groves and references contained therein.[3]

Resistance Measurements: Plug resistivities were measured from the inlet plug face to the outlet plug face using two-electrode techniques. The plug was electrically isolated from the coreholder body and other ground paths. Resistance was measured using a series equivalent circuit model on an LCR meter with a frequency setting of 1 kHz. Quality factors were very close to 0, indicating that the measurements were of nearly pure resistance.

TEST PROCEDURES

Plugs were of approximately 3.8 cm diameter and 7.6 cm length. Plug dimensions as well as air permeabilities and porosities were measured. The plugs were CT scanned. Homogeneous plugs of a wide permeability range were selected for relative permeability tests.

Similar procedures were used for each relative permeability test. A plug was placed between endpieces and was jacketed with Teflon heat-shrink tubing. These were inserted into a Viton rubber sleeve, which in turn was placed within the coreholder. Nitrogen permeability and X-ray measurements were recorded at ambient and 150°C with the test confining pressure imposed. Confining pressure was maintained as the coreholder temperature was reduced to ambient (24°C). The plug was saturated with brine. Backpressure (3,100 kPa) was imposed on the system. Permeability, resistivity, and X-ray measurements were recorded for the brine saturated sample at ambient and 150°C temperatures.

Production from the plug was switched from the normal flow port to the ceramic disk. Humidified nitrogen gas was injected at the inlet at a pressure 1,030 kPa greater than the system backpressure. Single-phase brine flow was produced through the ceramic disk until the brine saturation stabilized at about 20%. X-ray scans and resistivity measurements were recorded as the brine saturation decreased. After reaching the 20% residual brine saturation condition, production from the plug was switched from the porous disk to the flow port. The permeability of the sample to humidified gas was calculated from multiple rate and pressure measurements.

X-ray scans and resistivity data recorded while measuring the gas permeability were compared with data recorded during the desaturation process. Undersaturated gas led to sample dehydration. This situation could be inferred by (1) off-trend changes in resistivity versus brine saturation with little apparent change in brine saturations calculated from X-ray data and (2) pressure drop divided by gas rate ($\Delta P/Q_g$) values which decreased with increasing gas flow to a ratio similar to that measured for the dry sample. Use of oversaturated gas led to an increase in liquid saturation within the sample. This condition was inferred from (1) off-trend changes in resistivity versus brine saturations calculated from X-ray data and (2) $\Delta P/Q_g$ values that increased with time that were considerably greater than the ratio measured for the dry sample.

When vaporization or condensation effects were suspected, the gas humidifier temperature was adjusted, the plug was flooded with brine, and the desaturation and gas permeability measurement processes were repeated. After performing this fine-tuning process, vaporization or condensation effects were generally minimized in subsequent tests as long as the same coreholder and humidifier temperature settings were used.

When X-ray scan and resistivity results at the end of the gas permeability measurement agreed with those at the end of the desaturation process, the test proceeded

with two-phase flow measurements. Each measurement set included relative permeability data, resistivity, and X-ray scan data. Imbibition measurements were recorded for six two-phase gas/brine ratios ranging from 3,000/1 to 0.25/1. After measuring the brine permeability at a residual gas saturation condition, drainage flow measurements were recorded at gas/brine ratios ranging from 0.25/1 to 3,000/1 and finally with 1/0.

The brine pump and gas flow controllers were calibrated periodically throughout the testing program. Brine injection rates adjusted for density changes with temperature were used in brine permeability calculations rather than timed measurements of produced brine. This was done because both brine and water condensate from the gas accumulated in the gas-brine separator, making it difficult to distinguish between the two. Gas rates within the core plug were calculated by applying temperature, pressure, and humidity corrections to gas rates measured downstream from the backpressure regulator at ambient conditions.

TYPICAL RESULTS

Typical test results are shown in figures 4 through 7. Because the Norphlet Formation plug results were proprietary, results from a test on a Berea sandstone plug are presented for demonstration purposes. Test confining pressure and temperature conditions were 6.9 MPa and 24°C. The permeability of the sample to brine was 1,200 mD. Figure 4 shows changes in brine saturation versus time as the sample was desaturated through the porous ceramic disk segment of the endpiece. Figure 5 shows saturation profiles at various times during the desaturation process calculated from X-ray scan data. Resistivity index versus saturation results are shown in figure 6. Note that desaturation and imbibition resistivity data overlay each other. The drainage results were recorded at various times during the desaturation process as the sample was driven to residual brine under a single imposed pressure. Alternatively, we could have reduced the brine saturation within the sample with step changes in upstream pressure, yielding capillary pressure versus saturation data as well as resistivities. Steady-state gas and brine relative permeability versus saturation functions from imbibition cycle measurements are shown in figure 7. Single-phase measurements were used to obtain brine permeabilities at saturations greater than 0.8. As can be seen, both resistivity and relative permeability measurements are recorded during one test without changing test fixtures or disturbing the sample.

DISCUSSION

Achieving water-saturated gas proved to be one of the most challenging aspects of the project. The potential for accidentally dehydrating a plug sample is high. Figure 8 shows results from calculations of water content of fully saturated gas at three temperatures. The water content of saturated gas increases with temperature and decreases with increasing pore-pressure. About 6,000,000 standard cubic centimeters (6,000 standard liters) of gas is used during a typical steady-state gas-brine test in which the pore volume of the plug is about 18 cm^3. At 150°C and 3,100 kPa, approximately 900 mL of water is required to saturate 6,000 standard liters of an ideal gas with water vapor. The potential for dehydrating the plug is high because the water requirement for gas humidification is significantly greater than the volume of brine within the test plug. The water content of saturated gas decreases with increasing pressure. Therefore, simplification is possible for tests conducted at high pore pressures. As the gas pressure increases, though, it becomes increasingly difficult to achieve high gas-to-brine flow ratios during relative permeability tests. For this reason, test pore pressures have to coincide with limitations of available equipment, such as gas flow controllers. Because the water mass required to fully saturate gas increases with decreasing pressure, gas will dehydrate a plug to some extent as gas flows from the plug inlet to outlet. Maintaining

low flowing pressure drops through the core plug will minimize this effect, although capillary end effects tend to become more pronounced as flow rates are lowered.

Adding salt to water at a constant temperature causes the resulting brine solution to have a lower vapor pressure than pure water. For example, adding 5 gram-moles of sodium chloride to one liter of water at 100°C causes a reduction in the vapor pressure of the aqueous solution from 760 mm Hg to 617 mm Hg.[4] With respect to humidifying gas for a coreflood experiment, if gas is saturated with deionized or pure water at the same temperature as the coreflood experiment, and if the core plug contains a mixture of salt and water (brine), water vapor will condense from the gas in the plug pore space because of partial pressure reduction. To make the partial pressure of the water vapor in gas match the vapor pressure over the brine in the pore space, a water humidifier must be operated at a temperature lower than the brine temperature within the coreholder. If the temperature is higher, water condenses in the core plug. If the temperature is lower than the optimum setting, water molecules are vaporized from the brine in the core plug as the partial pressure increases to match the vapor pressure.

One would think that better moisture control could be achieved by humidifying gas through contact with brine instead of deionized or pure water. However, changes in the salinity of the brine in the humidifier can cause unexpected problems. Typically, humidifying gas with brine is accomplished by passing gas through a static column of brine at the test temperature. The salinity of the brine in the humidifier increases as water vapor is carried away with the gas. As the salinity of the brine in the humidifier increases, the vapor pressure of the brine is reduced, so the rate of water uptake within the humidifier by the gas decreases. Now, when this undersaturated gas enters the core plug, the higher vapor pressure within the core plug causes the gas to carry away additional water vapor as it passes through, dehydrating the sample. In severe cases, the sample can become plugged with salt.

Changes in brine salinity or in total dissolved solids can significantly affect measurements of key parameters such as saturation distributions and electrical resistivity. X-ray absorbers, like sodium iodide or potassium bromide, are often added to the brine to increase contrast between brine and gas phase X-ray absorption characteristics. Changes in fluid saturations within the rock are calculated from changes in the concentration of the X-ray absorber in the X-ray beam path. When the concentration of the absorber in the brine remains constant, the X-ray technique is very accurate for determining fluid saturations within rocks. When the concentration of the absorber in the brine changes, X-ray determined saturations become inaccurate. Likewise, resistivity techniques can be used to track changes in brine saturations as long as the brine salinity remains constant. By using both X-ray and resistivity techniques to track changes in fluid saturations, we were able to detect when incorrect gas humidification was either adding or removing water from the brine. Once this situation was observed, the humidifier was fine-tuned to achieve a proper balance between the partial pressure of the humidified gas and the vapor pressure of the brine at test conditions. The use of similar techniques is highly recommended to others who are involved in similar laboratory experiments.

CONCLUSIONS

The following are some of the conclusions from this work.

1. A newly designed endpiece provides for controlled desaturation to a desired level and the ability to continue with flow, resistivity, or capillary pressure versus saturation measurements without having to disturb the rock or cycle between ambient and test temperature and pressure conditions. The endpiece includes a system to allow for normal flow and a system for capillary desaturation through a porous ceramic disk.

2. The endpiece was used to desaturate plugs during tests at reservoir-conditions and to continue with steady-state relative permeability measurements without having to change endpieces or disturb samples.

3. Resistivity versus brine saturation functions measured while plugs were desaturated by porous ceramic disk techniques were similar to those measured during steady-state imbibition relative permeability tests.

4. Properly humidifying gas proved to be one of the most challenging aspects of the tests at elevated temperatures. The gas humidifier temperature had to be set lower than the coreflood temperature so that the partial pressure of the water vapor in the gas matched the vapor pressure of the brine at the coreflood conditions.

5. To minimize the potential for gas to hydrate or dehydrate a core plug during a flow experiment, the system backpressure should be set as high as practical, and the pressure drop through the sample should be kept as low as practical. These two constraints have to be balanced with respect to other limitations, such as target maximum gas/brine flow ratios and the sensitivity of available pressure measurement devices.

6. For tests with brine and gas, techniques to identify improper gas humidification need to be used. Improper humidification can seriously affect the accuracy of test measurements. Once identified, humidification problems can be minimized by changing experimental operating conditions, by fine-tuning the gas humidifier temperature, or by a combination of both.

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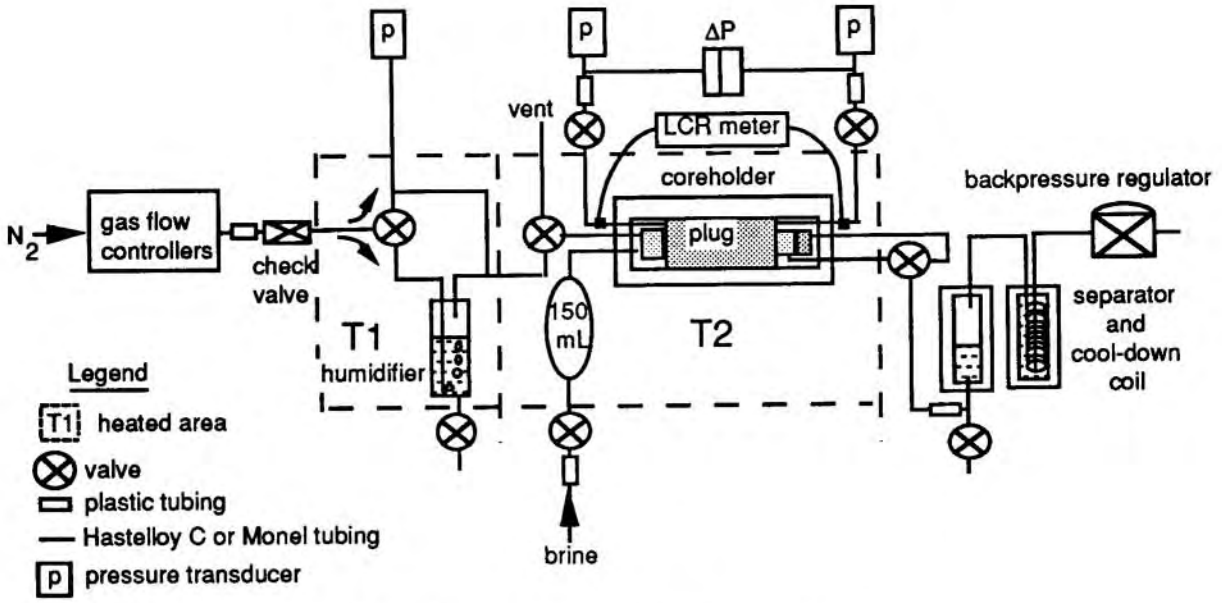

FIGURE 1. Flow system schematic

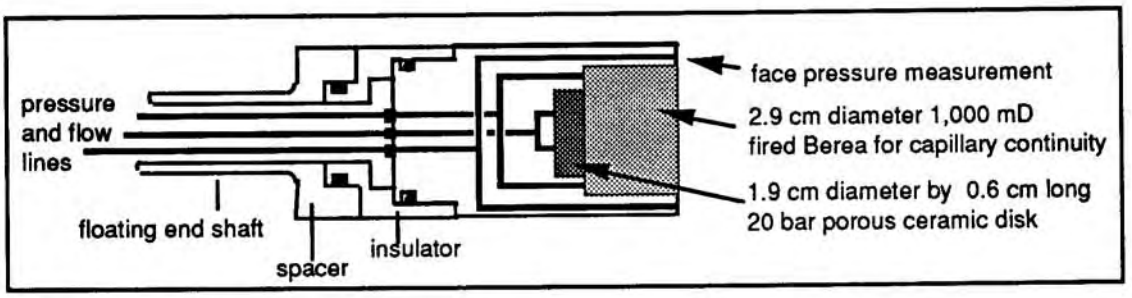

FIGURE 2. Endpiece with flow and porous plate desaturation features. The flow direction is from right to left.

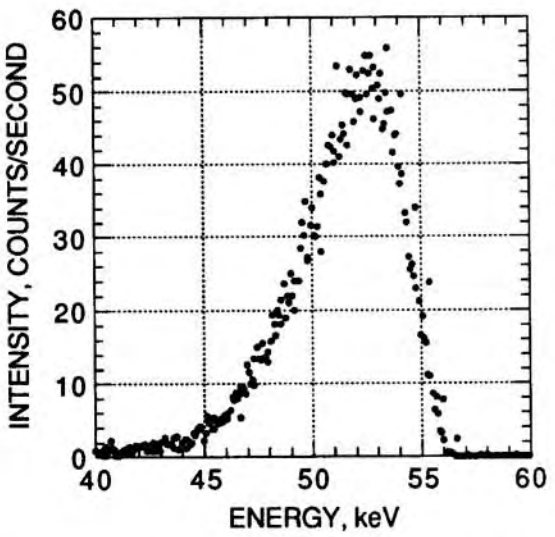

FIGURE 3. Typical X-ray scan for a brine saturated plug

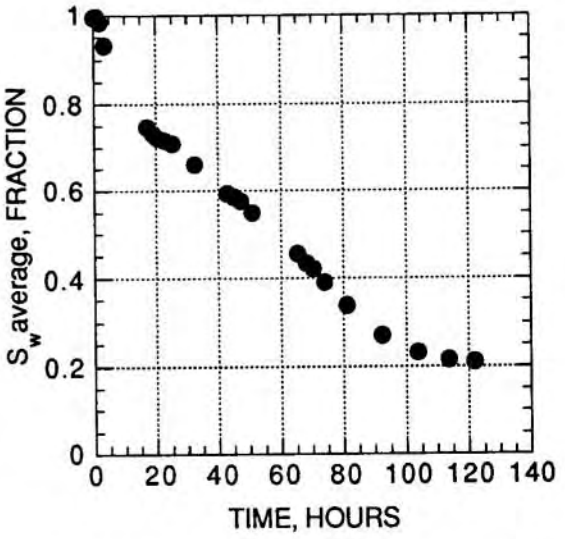

FIGURE 4. Porous disk desaturation results

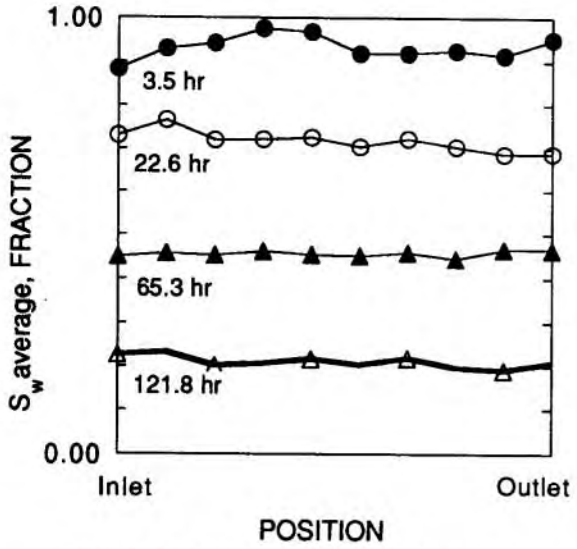

FIGURE 5. Saturation profiles during desaturation

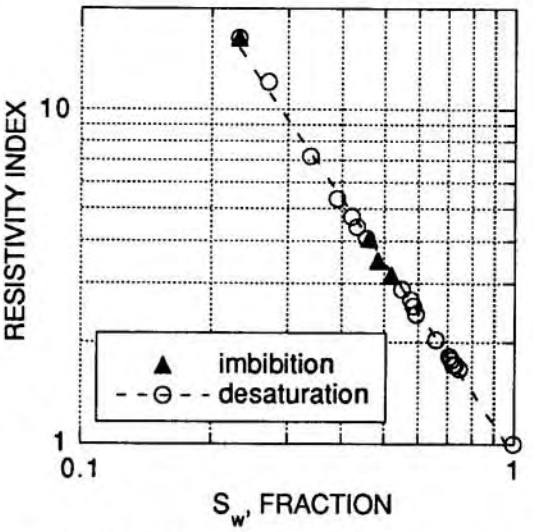

FIGURE 6. Resistivity index versus saturation results

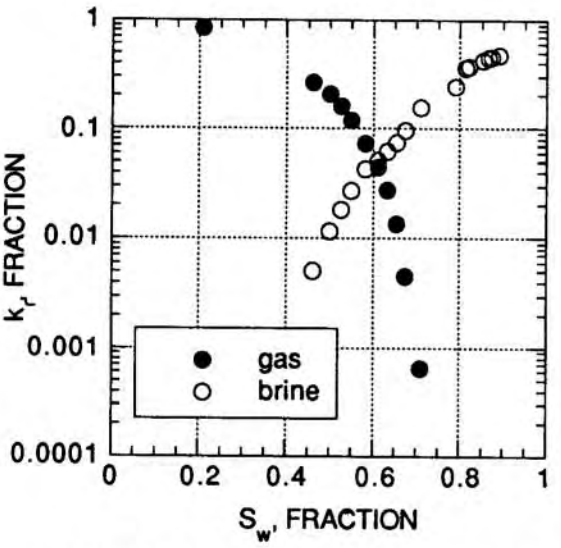

FIGURE 7. Imbibition cycle nitrogen gas and brine relative permeability results

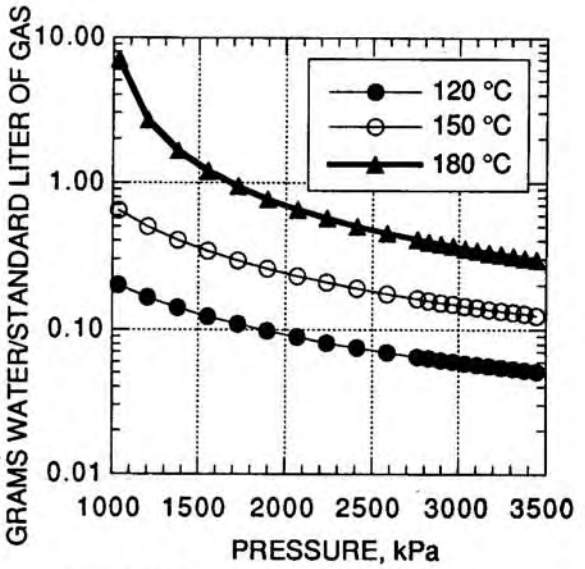

FIGURE 8. Calculated water content of saturated nitrogen gas at various temperature and pressure conditions