MISCIBLE DISPLACEMENT BY HIGH PRESSURE NITROGEN INJECTION

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INTRODUCTION

Primary oil recovery frequently results in oil recoveries of only 10 to 20 percent of the original oil in place. Several hydrocarbon miscible methods have been tested and proven successful as a secondary or tertiary method of oil recovery under certain reservoir conditions.^{1,2,3} Wide application of these methods has been limited by economic factors. Hydrocarbon miscible displacement had its beginning with high-pressure natural-gas injection more than 25 years ago.⁴ Complete miscible displacement results in the displacement of one fluid by another with the lack of a phase boundary between the two fluids. Natural gas injection was followed by other miscible displacement methods using LPG slugs, enriched gas, and CO₂. With the increasing cost of these gases, it appeared desirable to study the feasibility of oil recovery by use of high pressure nitrogen injection.

MECHANISM OF DISPLACEMENT

The requirements for direct miscibility between nitrogen and hydrocarbons are expected to be very high pressures for the normal reservoir-temperature range. Convergence pressure charts published by the Natural Gas Processors Suppliers Association⁵ indicate that for temperatures of 100° F to 400° F, the convergence pressure for pure methane and a mid-continent crude is in the range of 9,600 psi (Fig. 1). The convergence pressure for nitrogen and this crude would be expected to be still higher due to the inert characteristics of nitrogen. With these values in mind, consider as an example the ternary diagram in Figure 2. This diagram represents the percent composition of a three component system, nitrogen, C_1 - C_6 , and C_{7+} . The shape of the phase envelope is a function of the pressure on the system. At pressure P_1 , the combination of nitrogen and crude of 50 percent C_1 - C_6 , and 50 percent C_{7+} would establish equilibrium somewhere on the tie line shown. The equilibrium tie lines at pressure P_1 , for the most part, lie within the two phase region of the phase envelope. Equilibrium compositions falling within the two phase region are composed of some liquid and gas existing as two phases separated by an interface.

Increased pressure on the system would collapse the phase envelope as shown in Figure 2 for pressure P_2 . At pressure P_2 the portion of equilibrium tie lines within the two phase region is reduced as compared to pressure P_1 ; that is, fewer equilibrium compositions would fall within the two phase region. At some still-higher pressure, the convergence pressure would be exceeded and all points on the equilibrium tie line would fall outside the phase envelope. At this pressure the nitrogen and crude would be completely miscible. As previously stated, this would be expected to be in excess of 9,600 psi for some crudes.





In a flow or displacement process the miscible pressure can be much lower than that in a cell. As nitrogen is continuously injected into the system at some temperature and pressure, the process can be viewed in a step-wise fashion (Fig.3). Pure nitrogen comes into contact with reservoir crude of composition, say, 50 percent C_{7+} and 50 percent C_{1-} C_6 . The three components will establish some equilibrium point, say, R_1 on the tie line. This equilibrium composition lies within the two phase envelope and has some liquid and gas constituent associated with it which may be represented by gas of composition G_1 and liquid composition L_1 . Because of the difference in the velocity of gas and liquid components of equilibrium point R_1 , the gas moves ahead of its liquid counterpart and liquid L_1 is left to recontact more injected nitrogen. The process again repeats itself. Liquid L1 and pure nitrogen come into equilibrium along the tie line, say at R_2 . Again the equilibrium composition R_2 has some liquid and vapor constituents G_2 and L_2 associated with it. Gas G_2 and Liquid L_2 will again migrate at different velocities as injection continues, and liquid L₂ comes into contact with injected nitrogen. The process described repeats itself until the remaining liquid composition is reduced to, for the most part, C_{7+} and the lighter C_1-C_6 have been evaporated. This process describes the mechanism by which an enriched gas is produced in the reservoir



rock.

The previously described gas for each equilibrium step has moved ahead of its liquid to contact the original oil in place. Now consider the phase relations of this gas as it contacts the crude (Fig. 4). For this example, consider the initial gas to be nitrogen contacting again a crude of composition 50 percent C_1 - C_6 and 50 percent C_{7+} . Gas G_1 and the crude at some temperature and pressure again come to equilibrium at a point on the tie line. This equilibrium point, R_2 , lies within the two-phase region of the phase envelope and has some liquid and vapor constituents. The gas, G₂, moves ahead of its liquid counterpart in this stepwise fashion to contact original oil in place. Gas G₂ and the crude again come to equilibrium on the tie line at R₃. The process repeats until the gas moving ahead is miscible with the oil in place and no phase boundary exists. The gas and liquid compositions are equal and exist at the critical point on the phase envelope. Displacement of the reservoir crude may approach 100 percent by the leading portion of the miscible zone.

The percentage recovery of the oil in place is related to the amount of crude required for the development of the fully miscible zone. The rate of development of the zone is dependent on the initial



equilibrium relations between the nitrogen and crude and or proximity to the critical point on the phase envelope. The closer the initial equilibrium composition is to the critical point, the less crude is expected to be required to develop a fluid which is miscible.

The critical point on the phase envelope is related to the shape of the envelope itself. Increased pressure, as previously described, collapses the envelope and moves the critical point point closer to the first equilibrium tie line. This suggests that higher pressures would require fewer steps to be required in the development of an enriched gas which would be fully miscible with the oil in place.

Another factor affecting the initial equilibrium point in relation to the critical point is the initial composition of the crude. The change in the position of the tie line is shown in Figure 5. Crude A would be expected to require more crude or a greater distance to develop a fully miscible zone than Crude B.

Previous work done by Hutchinson and Braun with Atlantic Richfield in 1959³ describes what is called 'oil used up' in the development of the miscible zone as a function of the length of the porous media. Approximately 20 percent of the oil in place was required for the development of



miscibility with a 10 foot medium while approximately 8 percent was required in a 40 foot porous medium. The development of the zone with high pressure nitrogen would be expected to follow the same general pattern.

EXPERIMENTAL INVESTIGATION

Laboratory Apparatus

The laboratory apparatus used in this investigation of miscible displacement by nitrogen was designed to simulate reservoir temperatures and pressures. The schematic diagram, Figure 6, shows the overall laboratory equipment used. The coiled sand pack was 40 feet of 0.2-inch inside-diameter stainless-steel high-pressure tubing packed with 140-200 mesh sieved manufactured glass beads. Pore volume was determined by fluid injection into the evacuated sand pack. The exit line from the coiled pack was 0.075-inch inside-diameter tubing. The coiled sand pack was immersed in a constanttemperature oil bath, providing for controlled temperature of each run at 150°F. Back pressure on the system was held constant by the use of a dome pressured back pressure regulator designed for high pressure small volume flow. Production down stream was to a graduated cylinder where liquid volumes were measured. Produced gas was passed through a silica-gel adsorber and then to a wet test meter.





Samples of various crudes and field gas were recombined in a blender-type high-pressure cell from field fluids contained in the stock tank crude cylinder and gas cylinder. Gas is pressurized and displaced by water from the high-pressure water pump.

Experimental Procedure

Procedure for the miscible displacement test for the system shown in Figure 6 began with the recombination of the stock-tank crude sample with field gas. Recombination was accomplished by the use of a high-pressure blender-type mixing cell. Stock tank crude was introduced into the base of the 18,000-cc cell until full. Half of the stock-tank sample was then displaced by high-pressure gas from the gas cylinder. Mixing began while gas was continuously pressurized by water from the highpressure water pump and the gas was injected into the top of the mixing cell until a saturation pressure was achieved. With this sample, several series of runs could be conducted with a sample uniform in properties from run to run.

Prior to injection of the recombined sample into the coiled sand pack, the pack was cleaned with trichloroethane and then placed on a vacuum at 220° F for eight hours. Kerosene was then injected into the pack at the desired run pressure and pore volume determined. With the system then at constant pressure and above the bubble point, the recombined sample was displaced into the sand pack. After displacing 1.5 pore volumes of fluid from the core, formation volume factors and gas oil ratios were recorded for another one pore volume displacement. This was done at constant pressure held by the small-volume, high-pressure backpressure regulator. Constant rate of injection was maintained by the injection of mercury into the base of the recombined shaker cell. The temperature was constant at room temperature. The permeability was determined at these conditions.

With 2.5 pore volumes of fluid displaced from the core, saturation of the sand pack was considered complete. The oil bath was then heated to the desired run temperature and fluid expansion determined by withdrawing mercury from the shaker cell in sufficient volumes to maintain constant pressure. The core was then isolated when the run temperature was reached.

Nitrogen contained in the nitrogen cell was compressed to the desired run pressure by mercury injection into the base of the cell. At the desired pressure, nitrogen was then injected into the saturated pack. Injection rate was calculated to produce a nitrogen flood front advance of 1 foot per hour for the first hour and 3.5 to 4 feet per hour through the remainder of the run. Constant injection rate and constant pressure were then held on the system throughout the run. Produced fluids drop from run pressure to atmospheric pressure across the back pressure regulator and are collected in the graduated cylinder. Heavier liquid remains there and the volume is measured. Gas moves from the graduated cylinder to the pre-weighed silica gel adsorber. The change in the initial and final weight of the silica gel was used to determine the volume of intermediates adsorbed by the gel. The hydrocarbon recovered by the gel, considered in calculation of total recovery of each test, had an average molecular weight of 55. The gas passing through the adsorber was metered.

At the conclusion of each run, the core was isolated and brought to atmospheric pressure. Trichloroethane was injected into the core at 220° F and displaced by nitrogen at 2000 psig. The specific gravity of the produced fluid was determined and compared to a calibration curve of specific gravity vs. percent stock tank oil mixed with trichloroethane.

After the trichloroethane was displaced with nitrogen to breakthrough, the core was again brought to atmospheric pressure. A vacuum was placed on the pack for 8 hours at a temperature above 220° F. At the conclusion of the vacuum time,



the core was considered clean for the next test.

Recoveries

The oil recovery for a 54.4° API crude recombined with field gas to a solution gas oil ratio of 700 scf/bbl is shown in Figure 7. The ordinate represents percent recovery of oil initially in place vs. run pressure on the abscissa. All runs in Figure 7 were at a constant temperature of 150° F. Recovery was seen to range from 68 percent of oil originally in place at 3000 psig to 92.8 percent of oil originally in place at a run pressure of 5000 psig. Residual oil for the 5000 psig run was found to be 7.1 percent of the original oil in place by trichloroethane injection. Recovery of stock-tank oil in the graduated cylinder was 80.7 percent original oil in place with 12.1 percent recovery from the silica gel adsorber.





Cumulative gas is plotted versus cumulative oil in Figure 8. At nitrogen breakthrough, the slope of the line is almost vertical for practical purposes. The gas-oil ratio was almost constant for the 5000 psig, 150° F run until breakthrough, as shown in Figure 9.

Similar oil recoveries have been observed with crudes of 43[°] API gravity at higher temperatures and pressures.⁶ As stated previously, the intermediate composition of the sample and temperature and pressure affect the miscible qualities of the crude for high-pressure miscible displacement with nitrogen.

SUMMARY

The use of nitrogen to develop a high-pressure miscible displacement type recovery process has been studied in the laboratory. The system was designed to simulate reservoir conditions. Various nitrogen injection pressures were used to displace a recombined crude oil and natural gas sample. Injection rates, temperatures and pressures were held constant for each test. An oil recovery of greater than 90-percent was achieved at 5000 psi. A mechanism by which the miscible zone can develop is presented. The possible use of nitrogen to develop miscibility should be studied in those reservoirs where it is believed miscibility could be achieved with natural gas but natural gas is not available. Studies should also be made for those reservoirs where gas is available, but it is more desirable to sell the gas now than wait 10 to 15 years.

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