PERMEABILITY REDUCTION AND WATER IMBIBITION IN UNPROPPED FRACTURES IN CARBONATE-RICH SHALE

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ABSTRACT

Hydraulic fracturing of ultra-low permeability reservoir rock has revolutionized hydrocarbon production in the 21st century. High pressure completion fluids can imbibe into the rock matrix and alter geomechanical properties of the rock proximal to the fracture face. Imbibition of the fracture fluid can damage the formation reducing effective permeability but it can also improve hydrocarbon recovery near the hydraulic fracture. The geomechanical fluid sensitivities may result in rock softening thereby reducing fracture permeability and potentially resealing unpropped fractures over time.

A procedure was developed to investigate fracture face fluid sensitivities and quantify the amount of fluid imbibing into the fracture face. This workflow uses micro-CT and contrast agents to visualize the extent of imbibition, low field T_2 NMR to quantify saturation changes, and steady state liquid permeability measurements to investigate changes in the fracture permeability. This paper outlines the workflow for this procedure and experimental results of fluid/rock interactions are presented for brine on unpropped oil-saturated carbonate rich shale. The results show reductions in fracture permeability, and in some instances, the observed permeability diminishing to near the matrix permeability indicating a closed fracture. The results also show rapid countercurrent imbibition through the fracture face and average oil recovery by imbibition ranging from 31-46%.

INTRODUCTION

Ultra-low permeability hydrocarbon bearing reservoirs, such as the Marcellus source-rock play, have proven to be immense opportunities for production of oil and natural gas. In the United States, ultra-low permeability formations accounted for the majority of oil and natural gas production growth for the last decade. In all of these plays, hydraulic fracturing of horizontal wells was the critical technology making these formations economically viable.

During the hydraulic fracturing process, a portion of the fracturing fluid enters the formation via leakoff [1]. Some of the leakoff fluid mixes with formation fluids and is

produced as flowback water. Mixing is usually evidenced by changes in ionic concentrations of the produced water with time. It is often observed in some unconventional plays like the Eagle Ford that only a fraction of the fracturing fluid is recovered [2] meaning a portion of the fluid becomes trapped in the rock [3]. There are multiple driving forces that cause water to imbibe into the rock matrix: capillary action [4], osmotic flow [5], and hydration of clay minerals [6]. This paper focuses on systems with water-based completion fluids and hydrocarbon-saturated formations.

Water imbibition in ultra-low permeability reservoir rock has seen increased attention in recent years because of potential positive and negative effects it could have on recovery. For example, if the hydraulic treatment is performed with a water-based fluid and the formation initially contains mostly oil and gas, the water-based fluid will move into the rock matrix while oil and/or gas will enter the fracture, so that countercurrent imbibition increases the recovery of hydrocarbons [7]. Imbibition can potentially reduce hydrocarbon recovery by changing relative permeability near the fracture face. Roychaudhuri et al.[8] were one of the first to specifically investigate shale imbibition from a hydraulic fracture face. Roychaudhury et al.[9] showed that countercurrent imbibition rates in shale are a function of clay content and wettability.

Conductivity of the hydraulic fractures is important for the overall performance of a well. Completion fluids can have deleterious effects on the geomechanical properties of the rock which can result in reduced fracture conductivity [10]. Water-based fracture fluid influenced reductions in fracture conductivity were observed in propped and unpropped samples [11]. The reductions in fracture permeability were attributed to multiple causes; softening of the rock causing compaction and more proppant embedment, relative permeability alterations, and swelling of clay minerals. Kakkar [11] performed tests on unpropped fractured Eagle Ford core samples and showed how different completions fluids caused reductions in unpropped fracture perms with basic (pH > 7) solutions creating the greatest decrease. Kakkar also found that high clay content samples showed greater decreases in fracture permeability with increasing effective stress, suggesting that higher clay content samples exhibited greater fluid sensitivity. Zhang et al. [12] performed a propped-fracture fluid sensitivity study using Barnett shale in an API conductivity cell. Zhang et al. showed an 88% permanent decrease in fracture conductivity after brine was flowed through the fracture. They attributed the decrease in conductivity to the rock softening that allowed more proppant embedment.

This paper focuses on the worst-case scenario; unpropped fractures exposed to brine. Fractures were induced in cores with a shearing load frame and micro-Computed Tomography (micro-CT) was used to characterize the fractures. Steady-state permeability to decane measurements were done before and after breaking samples and changes in fracture conductivity upon exposure to brine were recorded over time. NMR T_2

measurements were used to monitor changes in saturation and micro-CT was used to visualize invaded areas after the test.

EXPERIMENTAL

Samples

Carbonate rich shale samples were used in this study. Plugs (2.54cm dia.) were cut from core, wrapped in cellophane and aluminum foil, and were preserved with a coating of wax. The trim ends of the samples were used for XRD mineralogy.

Fluids

Decane represented the oil phase in all experiments because its NMR hydrogen index is nearly the same as water. Brines doped with 50wt. % potassium iodide (KI) were used for the aqueous phase; the potassium iodide serves as the contrasting agent for CT scans. CT contrast tests with plugs saturated with different concentrations of KI brine were done beforehand and it was determined that high KI concentrations were necessary to provide sufficient contrast to visualize imbibition in shale. The small pores are beneath the resolution limits of the micro-CT used and given the relatively low porosity of these samples, more mineral is captured per unit voxel than fluid. With most minerals having greater X-ray attenuation than either brine or oil, compounded with the unlikeliness the imbibing fluid progresses as a continuous phase, large amounts of contrast agents were necessary to notice any appreciable differences in the images.

All brines were made with D_2O to eliminate the possibility of misinterpretation of the NMR T_2 distribution. The NMR instrument used for these measurements does not detect D_2O, therefore the amount of oil displaced from the sample was determined by the overall reduction in NMR signal intensity and confirmed with mass balance.

Workflow

All samples began the workflow with micro-CT screening with the broken or cracked samples being rejected (Figure 1). The micro-CT used was a Zeiss model 510 Versa with an auto loading stage. Samples were scanned at a resolution of 58µm/voxel at 140KeV/10W. This lower resolution scan was chosen to better cover the entire region of fractures and imbibed areas in the core plugs.

Acceptable samples were then dried in a vacuum oven for 24 hours at 80°C. Drying the samples was done in lieu of harsher solvent extractions to minimize the possibility of damaging the samples. After drying, the samples were checked with an NMR T_2 measurement to determine liquid content. The target signal volume for the dried samples is less than 0.2ml. If an average core plug is 2.54cm dia. by approximately 5cm long and is assumed to have a porosity of 8%, the total pore volume of the sample is roughly 2ml. Therefore drying to a residual signal of 0.2ml represents removing approximately 90% of

the native liquids. If the samples contained more than 0.2ml, they were returned to the drying oven for another 24h and the process was repeated until the samples were sufficiently dry, usually 48-72h total.

After drying, samples were pressure saturated with decane at 138 bar and 66°C. NMR T_2 measurements were acquired with a low-field spectrometer (GeoSpec2, Oxford Instruments) using standard data acquisition and processing methods (GITSystems Advanced, Green Imaging Technology).

Accurate measurement of matrix permeability in ultra-low permeability reservoir rock was a critical part of this investigation because without knowing the matrix permeability, it is impossible to determine if a fracture has closed. Permeability in unconventional reservoir rock can be measured by multiple methods; crushed rock permeability, pulse decay, and steady state[13]. The requirement for small particle size meant crushed rock methods were unsuitable. Pulse-decay permeability was an acceptable technique but was ruled out because interpretation of the data is not as straight forward as steady state methods. Further, since fracture permeability measurements were done steady state, it made sense to have a commensurate measurement for matrix permeability.

Steady state permeability measurements provided baseline matrix permeability. Decane was flowed through the plugs at very slow rates and the differential pressure was recorded. Core plugs were loaded into a heat-jacketed core holder. Chandler Engineering Q5000 syringe pumps controlled the flow of decane, typically less than 0.1ml/hr. The pumps were housed in a thermally controlled cabinet to mitigate the effects of temperature fluctuations in the room. Constant back pressure on the plugs of 35Bar was supplied by an accumulator. The net overburden pressure was maintained at 172Bar. Steady state permeability measurements were done at 66°C in order to help liberate residual waxes from the sample. Measurements were performed at multiple flow rates, two or more, and linear regression provided the permeability according to the Darcy equation and the methods outlined by McPhee at al.[14]

After the steady state measurements, the samples were dried at ambient conditions since plugs needed to be reasonably dry before they were fractured. The plugs were shrunk wrapped in Teflon and fractured in a load frame using offset plates to shear the plug. This method for shearing plugs usually created one continuous fracture longitudinally through the plug, the ideal fracture for this type of experiment. Shearing the plugs provided a fracture surface profile that was more representative of the fractures in the subsurface and far superior for these types of experiments compared with tensile failure fractures or saw cuts. Another micro-CT scan was done on the plugs ensuring fracture conformity; heavily fractured samples were rejected. Fractured plugs, still in their Teflon wrapper, were once again pressure saturated with decane.

The fracture permeability system was essentially identical to that used for the steady state decane permeability measurements except this system had larger capacity pumps. Back pressure was 35Bar, net overburden was 172Bar, and temperature 66°C. Fractured samples were loaded into the cells, in their Teflon wrappers, and decane was flowed through the crack at multiple steady rates to measure the fracture permeability. This was very similar to the steady state permeability to decane measurements with the exception that steady state pressure differential was now attained in hours not days. After the baseline fracture permeability was measured with decane, fluid flow was then changed to KI doped brine.

Unpropped fracture permeability to KI brine was measured over several days until steady state pressure drop was achieved. After KI brine flow, the samples were removed from their respective core holders and final NMR T_2 measurement was done. The volume of decane displaced from the plug was the difference in signal before/after the imbibition test. Micro-CT was done to get an idea of the extent of imbibition into the rock and examine the extent of changes to the fracture.

RESULTS AND DUSCUSSION

Porosities as measured by NMR T_2 relaxation ranged from 9.2% to 13.0% with values clustering around 10% (Table 1). XRD analysis on the samples provided the clay and carbonate content. Two different samples types were represented in this study, one that is high carbonate / low clay, the other low carbonate / high clay.

Table 1: Tabulated results for selected shale samples. SSK stands for Steady State Permeability.

Sample ID	XRD Carbonate	XRD Clay	Φ (p.u.)	Matrix SSK_{oil} (nD)	Fracture SSK_{oil} (nD)	Fracture SSK_{water} (nD)	% Oil Displaced
1	22.5	50.4	9.2	25.5	415.5	31.1	46
2	21.9	52.1	13.0	54.4	40266.7	3755.5	31
3	43.8	25.1	9.8	102.2	25004.3	504.6	33
4	42.2	29.1	10.2	63.4	3608.0	78.8	41

All the permeabilities of the measured sample were within one order of magnitude ranging from about 10-100 nD (Table 1). The steady state permeability measurements were performed at multiple rates and took about one week to complete.

After measuring matrix permeability, the plug samples were allowed to dry and were then sheared using offset plates a load frame. Micro-CT was used to analyze the fractures and verify that there was a single fracture in each sample extending from one end of the plug to the other. The two broken halves were pressure saturated with decane and reassembled in a core holder with no proppant or offset to keep the fracture open.

Fracture permeability was measured at steady state with decane at multiple rates over one day. Each flow rate reached steady state in about an hour. This paper does not address the relationship between increasing net confining stress and unpropped fracture permeability; net confining stress was maintained constant at 172Bar. Fracture permeability was independent of rock type.

The liquid flowing through the fracture was changed from decane to 50% KI in D_2O brine and pressure drop recorded over time. Changing the fluid in the fracture caused a significant decrease in the measured permeability. In two of the experiments, the fracture permeability decreased nearly to matrix permeability values signifying near total closure of the fracture. Micro-CT examination after the flow-through experiment was unable to identify the fractures that were created in samples 1 and 4 meaning the fracture aperture was reduced to less than the resolution of the micro-CT, 58µm. The rate that the fractures lost permeability was rapid and steady state was reached in a matter of a few days. There are two possible explanations for the loss of permeability when the fracture was exposed to water: 1) the fracture surface softened allowing the two halves to fuse, or 2) mineral dissolution of the contact points of the rock-rock interface caused the fracture aperture to shrink. Because of the quick decreases in fracture permeability, the former is more likely.

Micro-CT was able to identify regions within the plug samples that were infiltrated with the KI doped brine. Obtaining micro-CT images with noticeable fluid boundaries was difficult with two of the other samples not showing any. It was found that the brine rapidly dispersed nearly homogenously throughout these samples and immediate transfer of the sample to the micro-CT after the flow measurement was critical; reducing the total time the samples were exposed to the KI brine was also important for the same reasons. The left image in Figure 2 shows Sample 4 after fracturing and before fracture permeability measurements with decane and KI doped brine. The fracture in this sample is a hairline crack along bedding extending the entire length of the plug. After flowing decane and brine through the crack, the fracture is indistinguishable from the KI brine infiltrated matrix. The image on the right in Figure 2 is the same distance along the plug. The bright striations in the image on the right are regions infiltrated by KI brine. All tomograms were collected using the same acquisition parameters. There is noticeably more X-ray beam hardening, i.e. brightness along the edge of the sample, after exposure to the KI brine signifying a change in the density of the sample.

NMR T_2 relaxation measurements were done on the samples before and after the fracture flow-through measurements. The NMR measurement does not sense the D_2O that was used to make the brine, therefore, the change in saturations in the plug was determined by the loss of total signal intensity comparing NMR before and after flow-through measurements. The amount of oil displaced by brine imbibition is summarized in Table *1*Table 1. Samples that had greater oil displacement were also the ones where the fracture permeability decreased to near matrix permeability; there was a greater pore pressure gradient across the length of these samples allowing the brine to overcome some of the

capillary pressure restrictions. The exposure of the rock to the brine was only a few days and the speed and magnitude the saturations changed exceeded expectations.

CONCLUSIONS

This paper presents a new workflow for determining fracture fluid sensitivities. Novel to this workflow was the determination of the matrix permeability as a baseline comparison for fracture permeability, a new technique based on shear that created fractures with profiles more representative of those in the subsurface, and the use of micro-CT to image the extent of imbibition from the fracture face. The results of this exploratory study found that fracture face fluid sensitivity could adversely affect productivity and may allow unpropped fractures to seal. Several key observations were:

- Unpropped fracture permeability was reduced in all samples when exposed to brine. This is likely the result of the brine changing the mechanical properties of the shale. The shale softened and the two sides of the fracture annealed. Two of the four samples showed a near complete sealing of the fracture.
- Countercurrent imbibition into the fracture face occured quickly over the course of the experiment (2-3 days) with the invading brine displacing 31-46% of the original oil in the sample. The samples used were initially fully oil saturated. Samples that saw the largest amount of oil displacement also had the largest pressure gradients.
- Micro-CT can be used with an appropriate contrast agent like potassium iodide to image the infiltrated zones. The dispersion of the brine into the sample matrix occurs rapidly over the course of 2-3 days.

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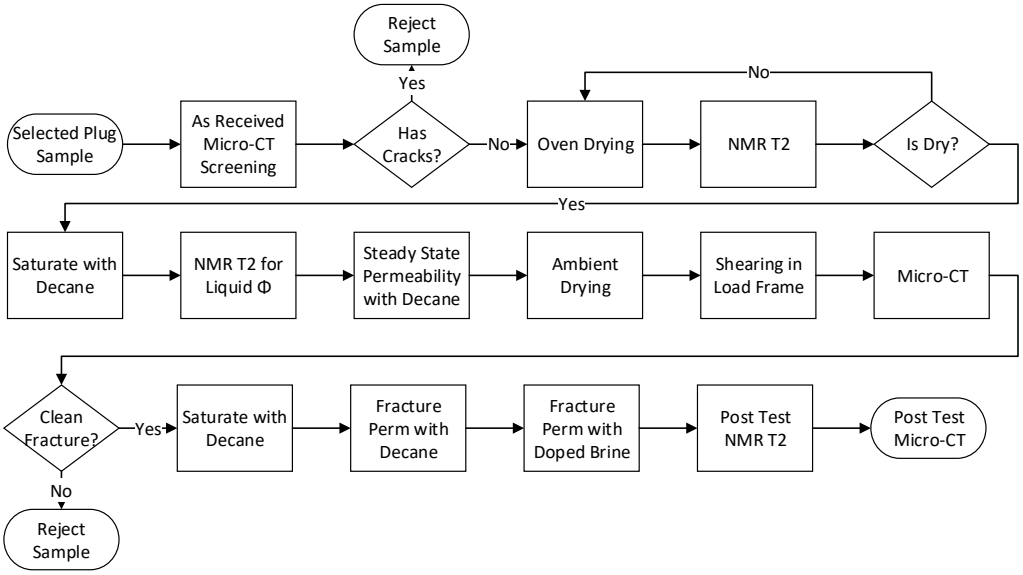

Figure 1: Workflow used for determining the effect of brine imbibition on fracture permeability.

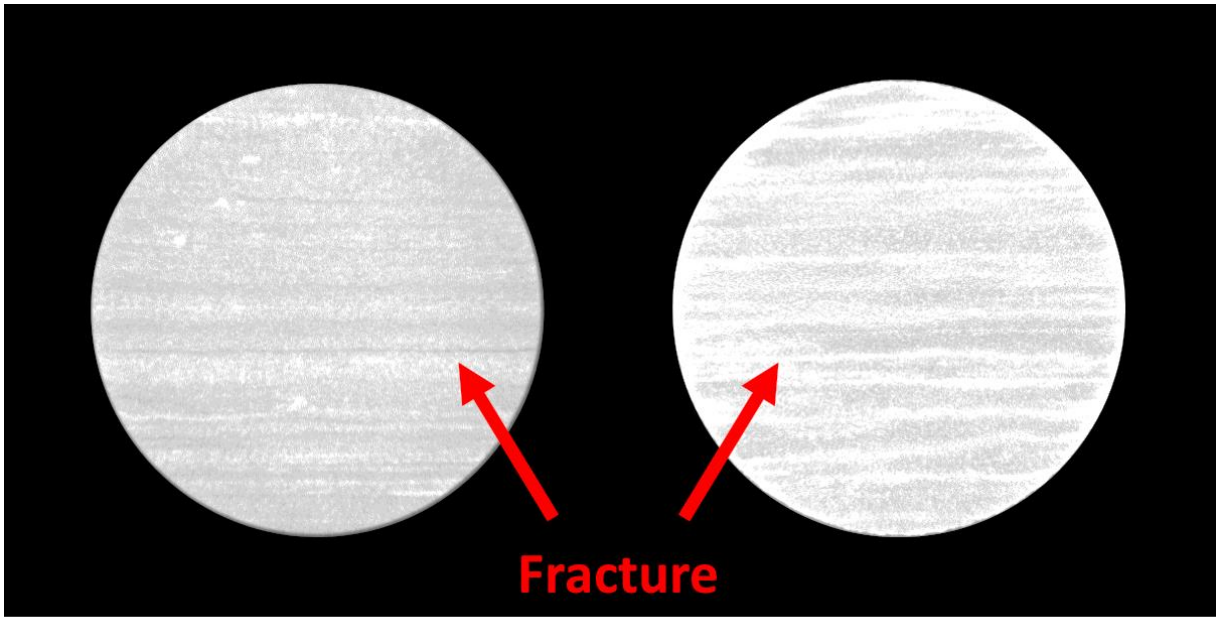

Figure 2: (left) Micro-CT tomogram of a fractured core plug 4V before fracture permeability measurement. (right) Micro-CT of the same core plug after fracture permeability measurement with decane and potassium iodine doped brine. The light colored striations in the sample are regions where the brine infiltrated. There is also a more pronounced beam hardening artifact signifying more X-ray attenuation around the edges of the plug. The fracture in this example completely sealed when exposed to brine. The resolution of both images is 58µm/voxel.