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Nanoparticle Networked Surfactant Micellar Fluids – Completion Fluids with Reduced Formation Damage

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Abstract

Polymer-based completion fluids are the most commonly used fluids for completion fluid loss and reservoir protection. These fluids can achieve high viscosities with low fluid loss rates for a wide range of reservoir temperatures and permeabilities. However, one weakness of polymer-based fluids is the permeability damage that occurs as a result of incomplete polymer residue removal from the pores of the near wellbore matrix.

Over the past two decades, surfactant-based fluids have been developed for gravel packing, frac-packing, and conventional hydraulic fracturing. These surfactant micellar fluids have viscoelastic fluid properties created by the overlapping and entanglement of thread-like micelle structures in brines. Surfactant micellar fluids exhibit viscosity-dependant leak-off control, and do not form filtercake on porous media. This type of leak-off is often inefficient and results in a significant amount of whole gel leak-off into the formation. For this reason conventional surfactant micellar fluids can only be used successfully for reservoirs with permeabilities up to about 200 md.

This paper will introduce a new low-damaging fluid loss control system based on nanoparticle associated thread-like micelles. Presented are newly developed, select nano-size inorganic crystals with surface forces that uniquely associate or “pseudo-crosslink” overlapping thread-like micelles into a 3D network. This unique fluid system works in KCl, $CaCl_2$, NaBr and $CaBr_2$ completion brines with fluid mix weights up to 14.4 ppg. Lab data is presented that shows pseudo-crosslinked thread-like micelle fluids have wall-building fluid loss control similar to crosslinked polymer fluids. The new nanoparticle-micelle association phenomena will allow surfactant-based fluids to be used for protecting reservoirs with permeabilities up to about 2500 md and temperatures up to about 300°F. Rheological tests are presented that show pseudo-crosslinked thread-like micelle fluids can achieve ultra-low shear rate viscosity above 1 million centipoise. Also introduced is micellar internal breaker technology, where the breaking agents preferentially reside within the thread-like micelle structures and will go wherever the micelles go for controlled and assured viscosity break. Core regain permeability data is also presented.

Introduction

Mechanical and chemical methods have been employed to control completion fluid loses into hydrocarbon-bearing reservoirs.[1,2] Of the mechanical devices, the most utilized appears to be downhole mechanical values. Most agree that mechanical fluid loss control should be the primary method to protect hydrocarbon-bearing zones and chemical methods for when tools cannot be used.

The most utilized chemical systems for reservoir protection have been polymer-based fluids. A wide variety of fluid loss control (FLC) pills have been developed and used.[2] It appear the most effective have been crosslinked polymer systems. Many of the polymeric systems also utilize particulates, such as specially sized salt and calcium carbonate to temporarily bridge the formation pores to further reduce rate of FLC pill leak-off. However, one main weakness with all polymer-based systems is the polymer residue they inherently leave behind, particularly in the form of filtercakes (i.e. concentrated polymer mass-layer created under pressure). The deposition followed by incomplete removal of polymeric filtercake often results in near-wellbore reservoir permeability damage.

Parlar et al. discuss the use of surfactant micellar fluids for FLC pills.[1] The surfactant-based technology available at that time had severe limitations, including restricted use to only low salinity and density mixwater brines. The performance verses cost of the surfactant-based fluid could not compete with the highly developed polymeric FLC systems, such as crosslinked HEC pills.

A new surfactant micellar fluid system called VES-PILL was developed and field evaluated from about 2000 to 2003.[3] The VES-PILL could be used in high salinity and density brines up to about 375°F. However, as much as 20% bw surfactant product was needed and very little low shear-rate fluid viscosity was achieved above about 300°F. Additionally, the fluid loss mechanism was still viscosity dependant, like previous surfactant micellar fluid systems. A second VES-PILL system was presented in 2006.[4] The authors note the leak-off mechanism of the VES-PILL was by the low shear rate fluid viscosity, and the surfactant-based pill does not have a wall-building mechanism for leak-off control.

This paper introduces a new surfactant-viscosified FLC pill system with enhanced performance and cleanup. The surfactant-based system utilizes patented and patent pending particles that are less than 50 nanometers in size, where up to 40% of the atoms are exposed at the surface of the particles. The select nanoparticles are inorganic crystals with unique surface charge properties. When placed into thread-like micelle fluids they "pseudo-crosslink" (i.e. appear to, but do not actually crosslink) the thread-shaped micelles into a 3D network. When the nanoparticle pseudo-crosslinked thread-like micelle fluid is placed under pressure and forced into porous media the networked thread-like micelles generate a pseudo-filtercake (i.e. have an appearance and similar property, but is not actually filter "cake"), exhibiting efficient wall-building leak-off control on porous media. Compared to crosslinked polymer systems, laboratory fluid loss tests show that nanoparticle pseudo-crosslinked thread-like micelle fluids have higher initial spurt-loss during the wall-building process but has similar leak-off rates once the pseudo-filtercake is formed.

This paper also presents patented and patent pending internal breaker technology, where the internal breaker resides within the thread-like micelles and will go wherever the micelles go. With this type of breaker mechanism the internal breaker concentrates with the tread-like micelles as the pseudo-filtercake develops. The internal breaker will also go with the thread-like micelles that leak into the reservoir matrix. The result is a self-breaking FLC pill that can be easily cleaned up. Since the surfactants that arrange in thread-like micelles are rearranged to form non-viscous spherical-shape micelle structures through the internal breaking process, the removal mechanism of pseudo-filtercake is completely different and more complete than the removal mechanism for crosslinked polymer filtercake. Presented in this paper is laboratory data on the rheology, fluid loss control, and cleanup for the new low-damaging fluid loss control system.

Tiny Solid Particles and Surfactants

In the past, addition of inorganic particles to viscous surfactant micellar fluids was perceived as counter-productive, in that the solid particles were viewed as pore plugging and thereby damaging to the reservoir permeability and their use would diminish the primary reason for using surfactant-viscosified fluids. However, two of the primary particles used for pseudo-crosslinking thread-like micelles are select ~35 nanometer size crystals. These tiny inorganic crystals are too small to plug the pores of reservoirs, even for 0.1 md permeability reservoirs. The gel-forming surfactants that generate thread-like micelles in brine have an average molecular weight of less than 600. Polymers used for FLC pills are typically greater than 1,500,000 in molecular weight. The size of the nanoparticles and surfactants used for the new surfactant-based FLC pill system are too small to bridge or plug the pores of reservoirs, unlike polymer-based systems.

Nanoparticle Pseudo-Crosslinked Micelles

A key parameter for the crosslinking of polymers is the degree of polymer-to-polymer overlap. The amount of overlap is typically dependant on the molecular weight of the polymer and polymer concentration within the fluid. Typically higher molecular weight polymers will overlap more easily than lower molecular weight polymers. Likewise, as polymer concentration increases the amount of polymer overlap increases. With more polymer overlap the polymers are easier to crosslink.

Similar to polymer chemistry, the length of tread-like micelles directly influences the degree of micelle-to-micelle overlap. The longer the thread-like micelles are the more intra-micelle overlap that can occur. Likewise, an increase in thread-like micelle concentration increases the amount of micelle-to-micelle overlap. **Figure 1** is a cryo-TEM photo from a study showing how thread-like micelles can be very long and overlap and one another.[5]

Figure 2 shows how nanoparticles can strongly associate thread-like micelles into a 3D network. The test fluid was 13.0 ppg $CaCl_2/CaBr_2$ with 2% bv gel-forming surfactant and 20/40 mesh-size ceramic particles. At the beginning of the test both samples were stirred to disperse the ceramic particles. The samples were then left static to observe particle suspension. After 10 minutes the test fluid without nanoparticles (bottle on the right) showed more than 90% ceramic particle settling. The nanoparticle pseudo-crosslinked thread-like micelle fluid (bottle on the left) showed no noticeable ceramic particle settling after 90 minutes under the same test conditions.

Similar to how crosslinking agents improve the performance properties of polymer-based fluids, select nanoparticles have been developed that improve the performance properties of thread-like micelle-based fluids. Many of these properties can be optimized for fluid loss pill utility.

Micelle Thermal Stability

Laboratory tests have showed that select nanoparticles can improve the thermal stability of thread-like micelles in brines. **Figure 3** is Brookfield PVS rheometer data at 275°F of thread-like micelles in 14.2 ppg $CaBr_2$ brine. The data shows how 0.014% bw nanoparticles can stabilize the micelles and resultant fluid viscosity of 1% bv gel-forming surfactant in brine, but without the presence of nanoparticles the thread-like micelles of 2% bv gel-forming surfactant are unstable at 275°F. For FLC pills the fluid viscosity upon heat-up is usually not important but how well the viscosity is retained over time. The ability to thermally stabilize thread-like micelles with a very small amount of "particles" is quite unique and cost effective.

Enhanced Viscosity Yield

The addition of ~35 nanometer size pyroelectric crystals to surfactant-viscosified fluids has showed significantly improved viscosity yield at moderate, low, and ultra-low fluid shear rates. **Figure 4** shows how 1% bv gel-forming surfactant with 0.046% bw pyroelectric nanoparticles has slightly more viscosity yield at moderate shear rate (100 sec-1) than twice the gel-forming surfactant (2% bv) without nanoparticles. Low shear rate (2 sec-1) tests showed an approximately two-fold increase in fluid viscosity by the addition of 0.046% bw nanoparticles to 2% bv gel-forming surfactant fluid, as shown is **Figure 5.** The low shear rate measurements were taken after the fluids were static for 30 minutes at 150°F. **Figure 6** shows that up to a 10 fold viscosity increase can be obtained at ultra-low shear rates for surfactant micellar fluids containing nanocrystals. The viscosity measurements in **Figure 6** were generated using a cone-plate oscillatory rheometer and the viscosity is shown in Poise. The centipoise (cps) values at 0.001 sec-1 shear shows the 2% bv gel-forming surfactant fluid is about 200,000 and with 0.077% bw pyroelectric nanocrystals is about 2,000,000. The rheology test data shows that a small amount of nanoparticles pseudo-crosslink thread-like micelles into a more viscous network, similar in respects to the addition of crosslinker to polymeric fluid.

Pseudo-Filtercake Leak-off Control

Laboratory fluid loss tests were performed using 0.25 in. by 2.5 in. Aloxite ceramic discs having 400 md permeability. The tests were to compare the wall-building efficiency of thread-like micellar fluid with and without nanocrystals. Tests results in **Figure 7** show that without nanocrystals the leak-off rate is very high and shows only the conventional viscosity-dependant leak-off control mechanism. The fluids with nanocrystals showed a moderately high spurt followed by a significantly reduced rate of fluid leak-off indicating development of a viscous fluid layer of pseudo-filtercake on the face of the ceramic discs. The fluid with nano-size pyroelectric crystals generated a slightly more efficient pseudo-filtercake layer than the fluid with product Cx nanoparticles. The photo on the left in **Figure 12** shows the pseudo-filtercake developed during the pyroelectric nanocrystals test. The photo on the left in **Figure 13** shows crosslinked polymer filtercake. The photos show that pseudo-filtercake has similarity to crosslinked polymer filtercake. However, the degradation mechanism to remove the polymeric-filtercake is significantly different and often problematic. It should be noted that viscous surfactant-based fluids would not have much importance if polymeric filtercake was easily and efficiently removable.

Recent fluid development work has showed utilization of low amounts of viscous mineral oils improves the wall-building leak-off efficiency of nanoparticle pseudo-crosslinked thread-like micelle fluids. **Figure 8** shows the effect of 0.6% bv Product 5N mineral oil on pseudo-filtercake. A decade ago thread-like micelles in brine were perceived to break upon contact with hydrocarbon fluids.[6-7] However, lab tests have showed this is not always the case.[8] The data in **Figure 8** shows the hydrocarbons in 5N mineral oil do not induce thread-like micelle degradation upon contact but rather the presence of mineral oil reduces the fluid leak-off over time. The deposition of nanoparticle pseudo-crosslinked thread-like micelle fluid layer appears to hinder the mineral oil from going through the pseudo-filtercake and the mineral oil appears to concentrate within and in contact with the thread-like micelles composing the pseudo-filtercake.

Like the mechanism to develop pseudo-filtercake is quite different than the deposition of polymeric filtercake, the mechanisms of cleanup are also very different.

Molecular Level Breaking Mechanism

Polymer-based FLC pills do not have an internal breaking mechanism where the breaker stays with the polymer at the molecular level and goes wherever the polymer goes to degrade polymer chains back to monomer units, like HEC polymer reduced back to glucose. Even with the use of enzymes or high concentrations of oxidizers, polymer degradation to monomers is very incomplete and residual polymer mass still remains.

Surfactant-viscosified fluids in the past were perceived as not requiring internal breakers. The polymer free systems were suspected to cleanup by contact with reservoir fluids, particularly by contacting reservoir hydrocarbons.[6-7] However, operators have become aware that poor fluid cleanup occurs too frequently in the field. A recent study shows that not all reservoir hydrocarbons will breakdown viscous thread-like micelles in brine by simple contact, but that the two fluids are immiscible and sufficient mixing energy is required and often not provided within the reservoir pores.[8] Additionally, surfactant micellar fluids have very high viscosity at very low-shear rates. Without the use of internal breakers, unbroken surfactant micellar fluids will require higher formation pressure to displace from the pore matrix of the reservoir and incomplete cleanup may occur. **Figure 9** shows surfactant-viscosified fluids without internal breaker will retain their high viscosity at low-shear rates and how internal breaker can reduce the low-shear rate viscosity back to brine-like viscosity without the need to contact and mix into reservoir hydrocarbons. **Figure 10** shows the initial flowback pressure displacing thread-like micelle fluid from 6 in. long Berea core. The test without internal breaker required a substantial amount of pressure to initiate flow and to push the viscous fluid from the core pore matrix. However, the amount of pressure required to cleanup partially broken surfactant-gel fluid was substantially reduced, and for completely broken fluid very little cleanup pressure was required.

Internal breakers have been recently developed for surfactant-viscosified fluids where the breaking compounds reside within the thread-like micelles and will go wherever the micelles go.[9] **Figure 11** is an illustration of how internal breaker compounds are distributed in the thread-like micelles and upon activation breaks the fluid viscosity by rearrangement of the thread-like micelles to non-viscous spherical shaped micelles and nanoparticles. With this mechanism the internal breaker becomes a chemical and structural part of the thread-like micelles. During fluid loss into porous media the internal breaker stays within the tread-like micelles as the micelles concentrate during pseudo-filtercake development. With internal breaking agents that are within each thread-like micelle on the molecular level, very efficient and more assured thread-like micelle degradation can be achieved. The internal breaker will also go with the micelles that leak into the reservoir matrix.

One patented internal breaker technology is the use of organic compounds that auto-oxidize. In their original form, the organic compounds have high compatibility with thread-like micelle structures in brine. The mechanism of how they work as internal breakers is based on the auto-oxidation of select chemical bonds. Several organic materials have been found that auto-oxidize at different rates for use from 80°F to about 300°F. When the select bonds go through the process of auto-oxidation the organic compound becomes fragmented into hydrocarbons that can degrade and change the structure of thread-like micelle by simple surfactant rearrangement into non-viscous spherical shaped micelles. When the organic materials are added to surfactant-viscosified fluids the hydrophobic organic material interacts with the surfactant hydrocarbon tails and during initial mixing they are dispersed at the molecular level within each thread-like micelle. The rate of auto-oxidation of the organic material increases with fluid temperature and slows with increase in fluid salinity. By optimizing the type and amount of organic material, along with salinity, the viscosity of the FLC pill can be controllably reduced in a few hours or up to several days. This micelle rearrangement breaking mechanism is more efficient than current mechanisms for polymer chain degradation.

Fluid Loss Pill Cleanup

Fluid loss control pills are routinely used without the addition of internal breaker since by design the largest portion of the pill fluid remains within the wellbore. Typically the pills are used for a period of time and then displaced out and a breaker solution is spotted in the wellbore to breakdown and cleanup the remains of the pill, particularly the filtercake and any particulate bridging agent. If necessary, the same cleanup method can be used with the nanoparticle pseudo-crosslinked thread-like micelle-based FLC pill. Pseudo-crosslinked micelles are much easier to degrade than crosslinked polymer filtercake.

The photo on the left of **Figure 12** shows the deposition of pseudo-filtercake on 400 md Aloxite ceramic disc after 70 minutes of leak-off. The pseudo-filtercake is a thin deposit of nanoparticle associated thread-like micelles containing internal breaker. The photo on the right of **Figure 12** is Berea sandstone wafer discs and 20/40 mesh-size bauxite particles after a regain conductivity test. During the initial portion of the conductivity test nanoparticle pseudo-crosslinked thread-like micelle fluid containing internal breaker was injected into the bauxite particles and forced to leak-off into the Berea sandstone wafers to develop pseudo-filtercake on the Berea wafers. The Berea wafers had approximately 400 md permeability. The test cell was then shut-in for the internal breaker to auto-oxidize and break the thread-like micelle fluid in the bauxite particles and the pseudo-filtercake deposited on the Berea wafers. The regain conductivity was then measured and the test cell was cooled and opened up for visual inspection. As the photo on the right of **Figure 12** shows, the internal breaker completely degraded the pseudo-filtercake on the Berea wafers and from the ceramic particles.

The cleanup of nanoparticle pseudo-crosslinked thread-like micelle fluids from 1 in. by 6 in. Berea cores were performed. Test A was with ~50 md Berea core and the Test B was with ~500 md Berea core. The results are shown in **Figure 13**. The data is normalized regain permeability and shows nanoparticle pseudo-crosslinked surfactant-gel cleaned up to gas (N_2) and KCl brine, leaving very little permeability damage.

Figure 14 has been added to show how polymeric fluids, when viscosity broken, may leave residual polymer-mass. The photo on the left shows crosslinked polymer filtercake deposited on 400 md Aloxite disc. The middle photo in **Figure 14** shows how concentrated crosslinked polymer can be “broken”, but retains a brittle bulk-mass property that can be difficult to cleanup. The photo on the right of **Figure 14** shows a typical 0.42% bw crosslinked polymer fluid after being “broken” by a high amount of sodium bromate oxidizer at a test temperature of 250°F. The photo shows how polymer typically does not degrade into readily producible monosaccharide, disaccharide, or oligosaccharide units.

Conclusions

1. Thread-like micelles generated by 1% bv gel-forming surfactant in 14.2 ppg $CaBr_2$ brine can be thermally stable at 275°F with 0.014% bw nanoparticles.
2. Addition of 0.077% bw ~35 nm pyroelectric particles can increase the 0.001 sec-1 fluid viscosity of 2% bv gel-forming surfactant fluid from about 200,000 to about 2,000,000 centipoise.
3. Nanoparticle pseudo-crosslinked thread-like micelle fluid will form pseudo-filtercake on porous media that exhibits efficient wall-building leak-off control.
4. The auto-oxidation of select organic material is one internal breaker mechanism that can be used to controllably rearrange thread-like micelles into non-viscous spherical micelles in brine.
5. Additional laboratory tests are needed to further compare the performance and cleanup properties of polymeric FLC pills with FLC pills based on nanoparticle pseudo-crosslinked thread-like micelle fluids containing internal breakers.

Acknowledgement

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Nomenclature

BHP = *Bottomhole Pressure*
BHST = *Bottomhole Static Temperature*
bv = *By Volume*
bw = *By Weight*
cps = *Centipoise*
ºF = *Degrees Fahrenheit*
ºC = *Degrees Celsius*
gal = *Gallon*
in. = *Inch*
md = *Millidarcies*
ml = *Milliliters*
ppg = *Pounds Per Gallon*
pptg = *Pounds Product Per 1000 Gallons Of Fluid*
sec $^{-1}$ = *Reciprocal Seconds*
sq ft = *Square Feet*

SI Metric Conversion Factors

cps	*x 1.0**	*E-03*	=	*Pa.s*
lbs	*x 4.535924*	*E-01*	=	*kg*
md	*x 9.869223*	*E-04*	=	um^2
ºF	*(ºF-32)/1.8*		=	*ºC*
psi	*x 6.894757*	*E+00*	=	*kPa*
ft^2	*x 9.290304**	*E-02*	=	m^2
lbs/ft	*x 1.355818*	*E+00*	=	*Nm*
inch	*x 2.54**	*E+00*	=	*cm*
gal	*x 3.785412*	*E-03*	=	m^3

** Conversion is exact*

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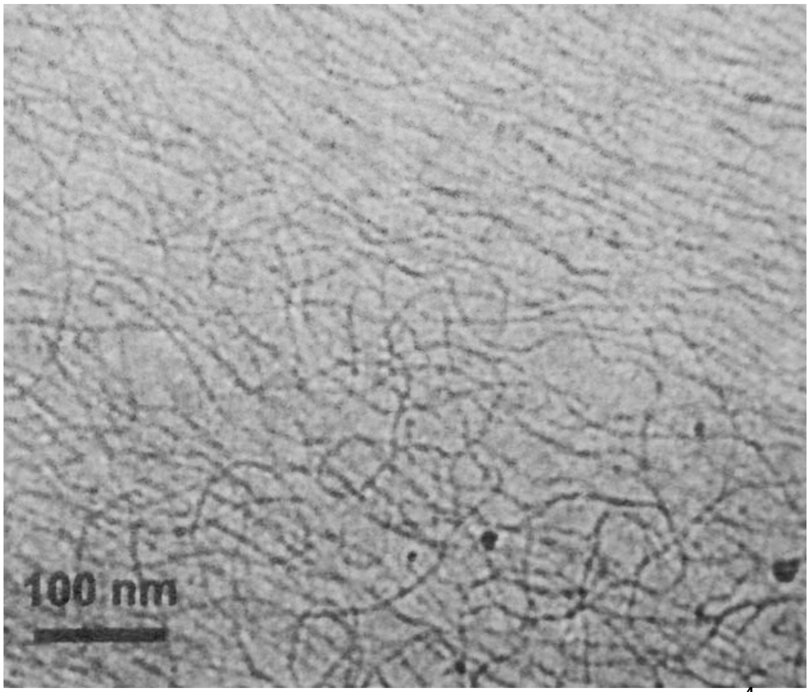

Figure 1. Cryo-TEM image of thread-like micelles (Kumar et al.[4]). Upper section shows micelle stretch orientation created during cryo-TEM sample preparation.

Figure 2. Photo shows ceramic particle suspension after 90 minutes. Both test bottles have 2% bv gel-forming surfactant. Test bottle on left contains 0.077% bw nanoparticles and shows strong pseudo-crosslink of thread-like micelles.

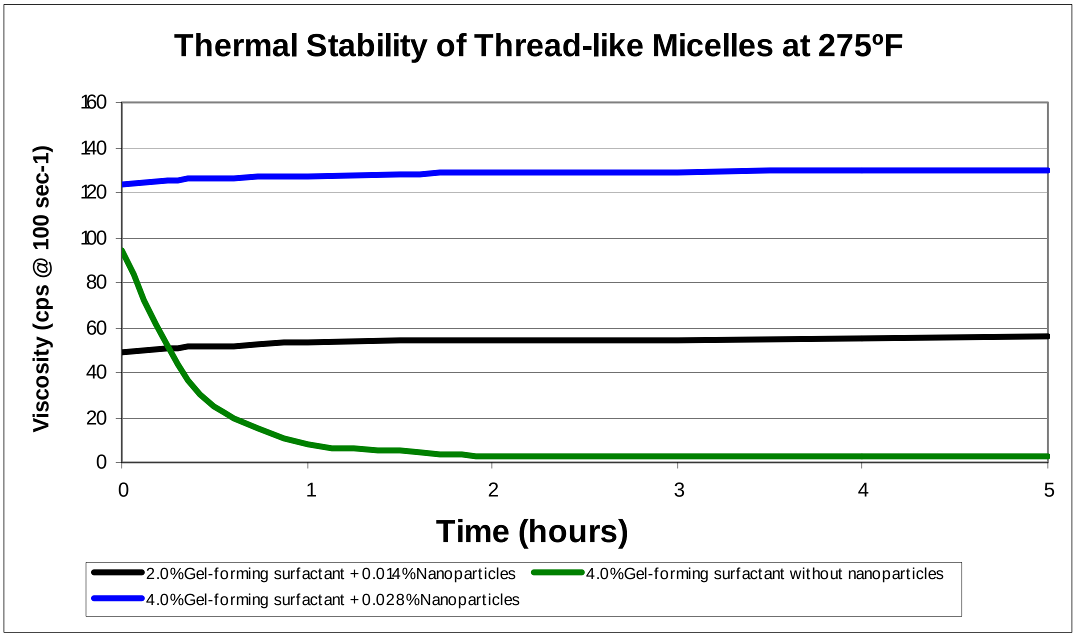

Figure 3. Low concentration of nanoparticles improves the thermal stability of thread-like micelles in 14.2 ppg $CaBr_2$ brine.

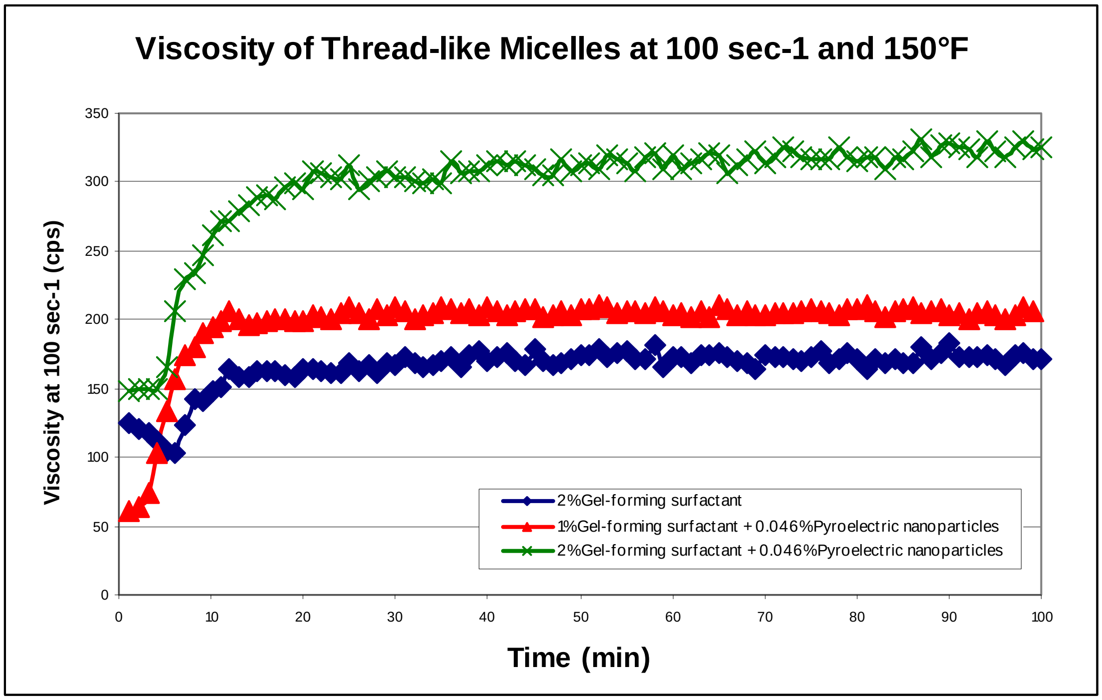

Figure 4. Addition of 35 nm pyroelectric particles improves fluid viscosity of thread-like micelle fluid at 100 sec-1 shear.

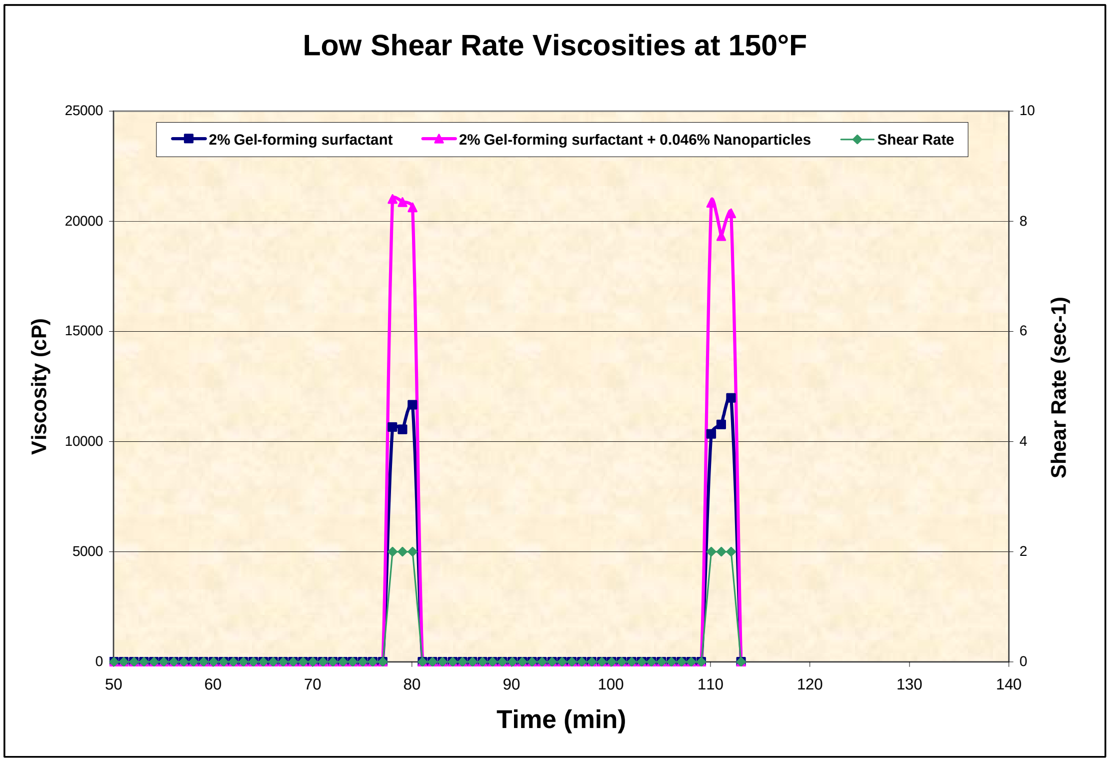

Figure 5. Low shear rate viscosity of surfactant-viscosified fluids after 30 minutes of no shear. The 2 sec-1 shear rate measurements show the sample with nanoparticles has improved thread-like micelle associations.

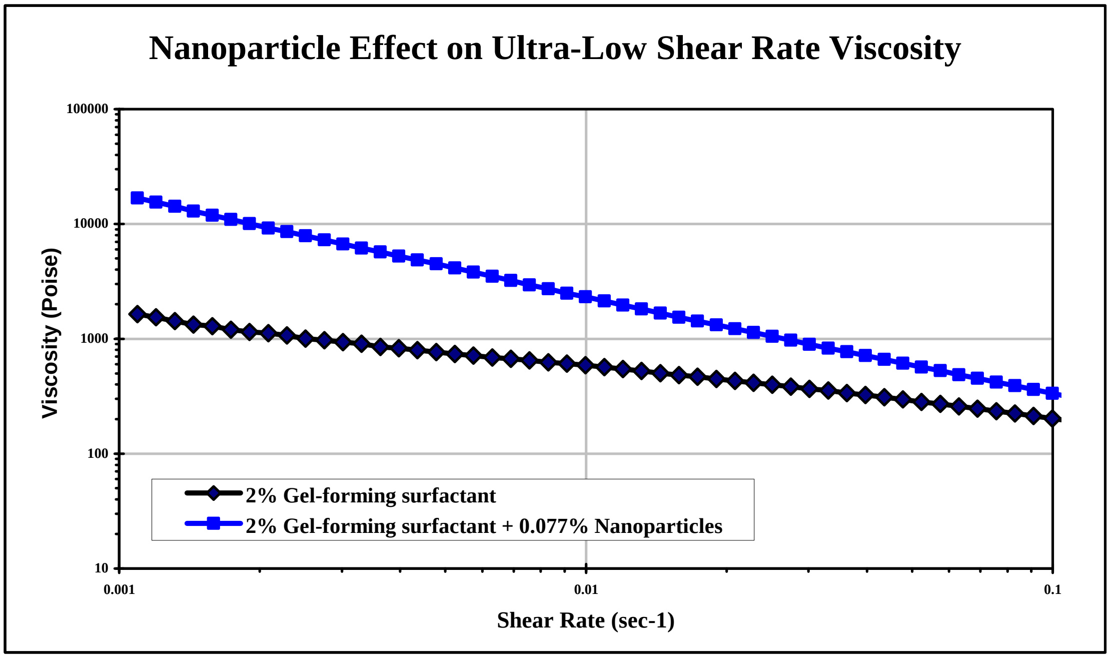

Figure 6. Ultra-low shear rate viscosity in Poise for thread-like micellar fluids with and without nanocrystals.

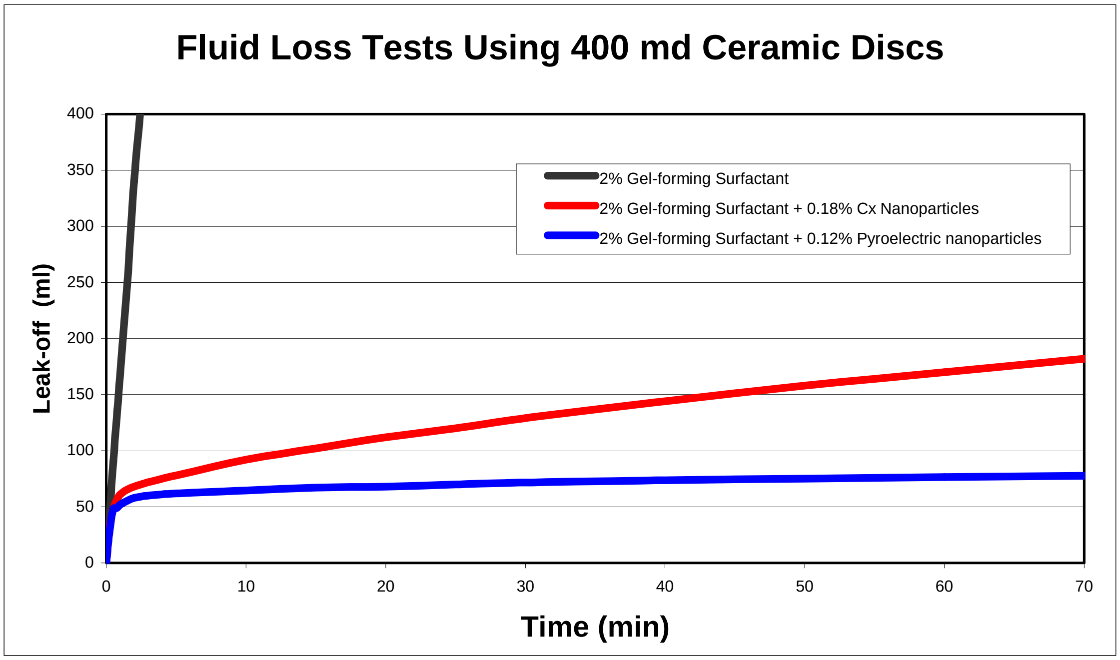

Figure 7. Fluid loss comparison of thread-like micelle fluid with and without ~35 nanometer size particles. The abrupt change in leak-off rate after 50 mls shows pseudo-filtercake development with wall-building fluid loss control. The initial spurt of pseudo-crosslinked micelle fluids is higher than for crosslinked polymer systems.

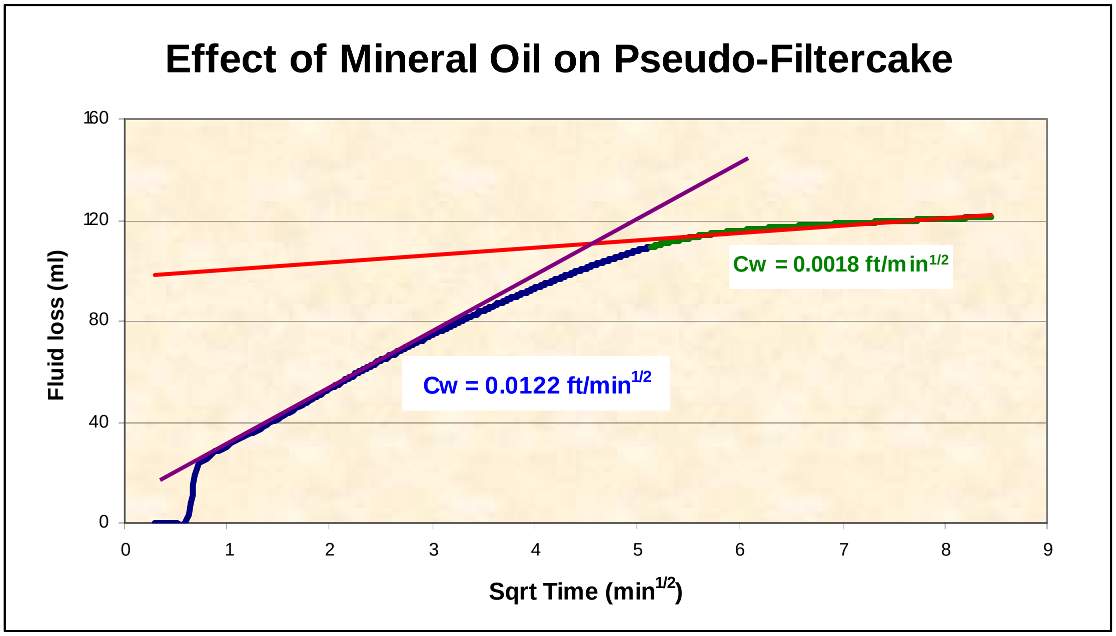

Figure 8. The use of low amount of viscous mineral oil does not break gel viscosity but improves the pseudo-filtercake efficiency.

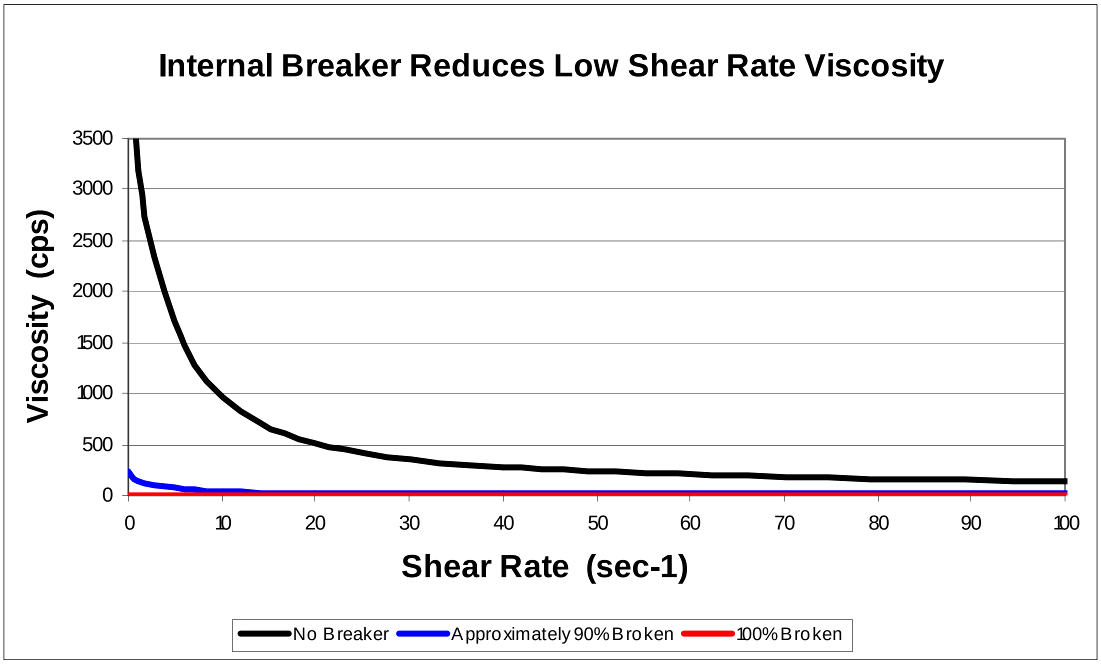

Figure 9. Effect of internal breaker on low shear rate viscosity for improved fluid cleanup.

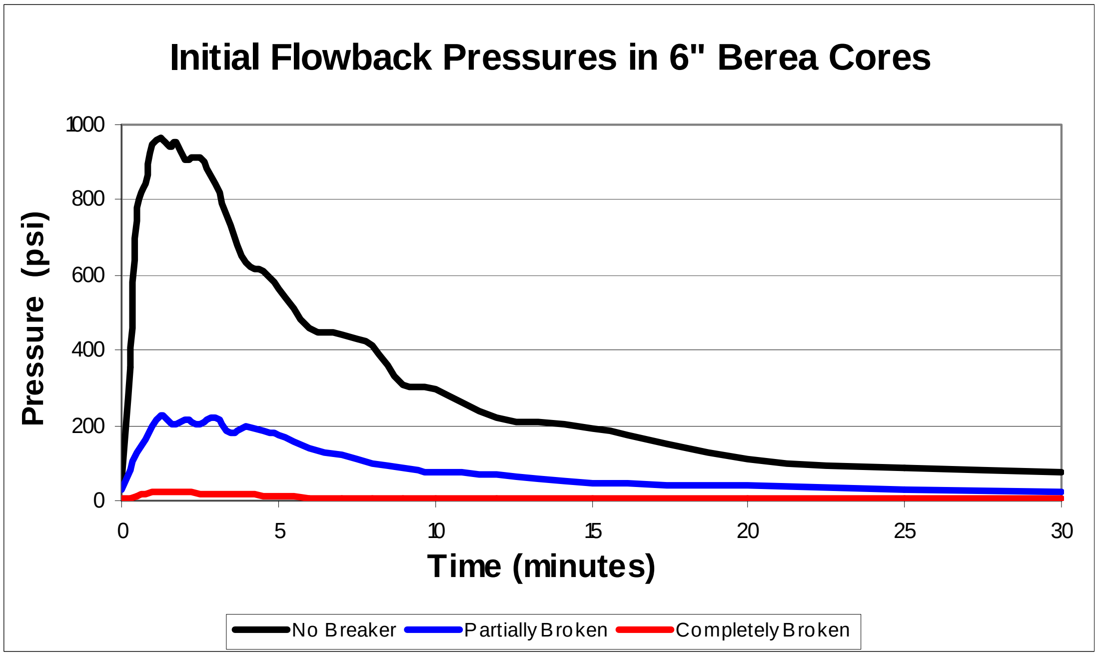

Figure 11. Internal breakers reduce flowback pressures required to displace surfactant-viscosified fluids from porous media.

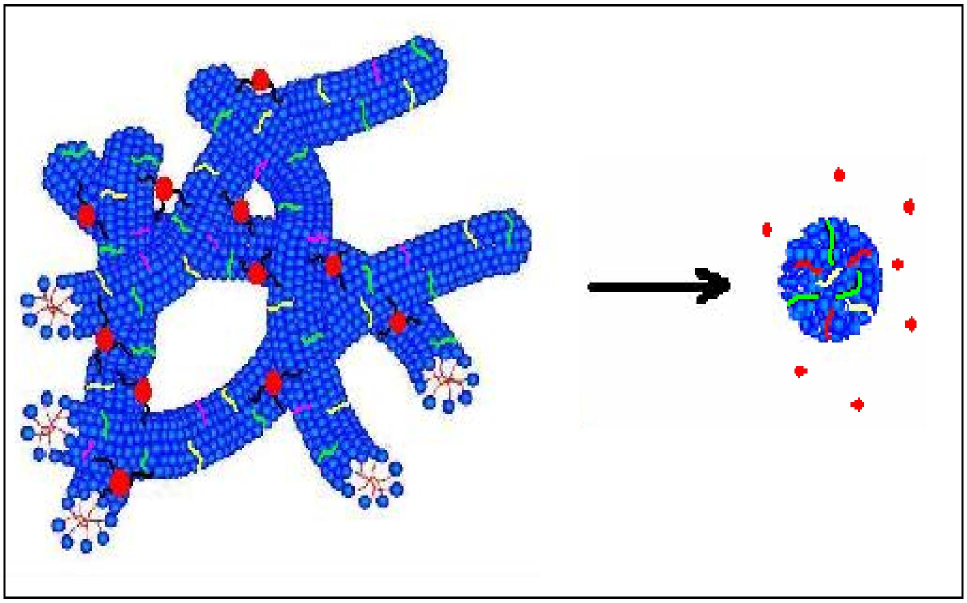

Figure 11. Illustration of internally breaking nanoparticle pseudo-crosslinked thread-like micelles by simple micelle rearrangement.

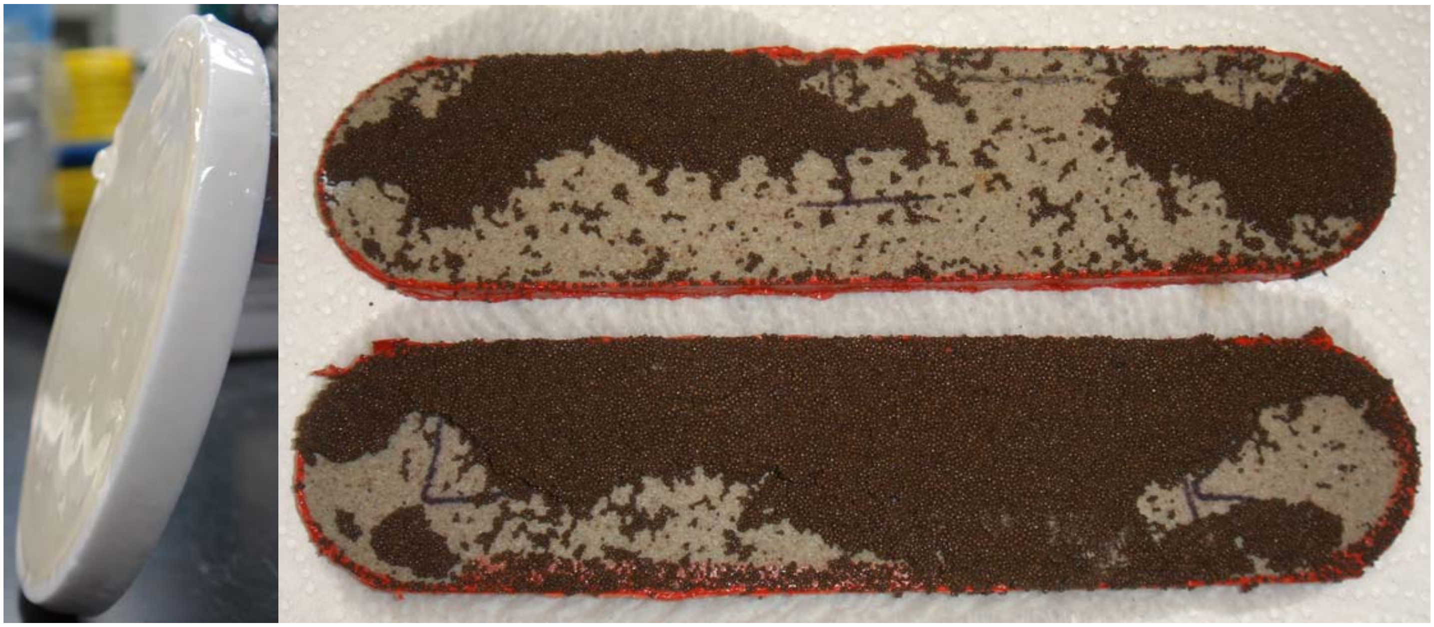

Figure 12. Photo on left is pseudo-filtercake on Aloxite disc after 70 minute fluid loss test. Photo on right is the Berea core wafers and ceramic proppant after a regain conductivity test, showing complete removal of pseudo-filtercake from the Berea core face and from ceramic particles by internal breaker.

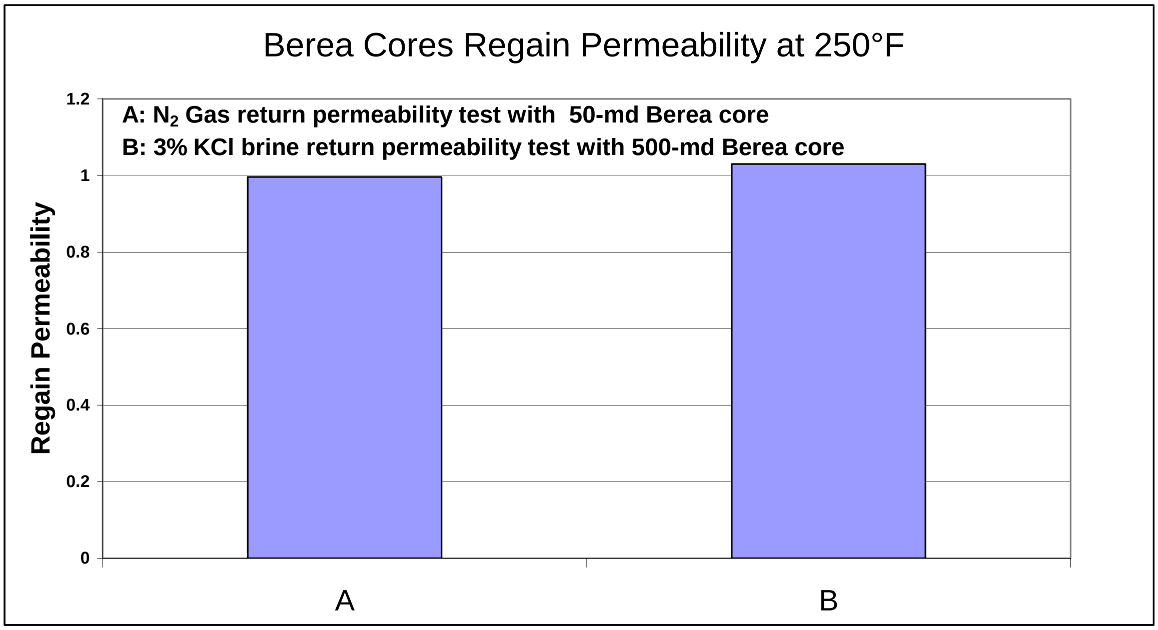

Figure 13. Core regain permeability tests for nanoparticle associated thread-like micellar fluid.

Figure 14. Photo on left shows a thin layer of crosslinked polymer filtercake. The middle photo is a concentrated mass of “broken” crosslink polymer, showing polymer residue is often brittle and easy to cut but can be difficult to cleanup. The photo on right is “broken” crosslinked polymer fluid showing polymer does not completely degrade to monomers.