

Critical Considerations for Successful Hydraulic Fracturing and Shale Gas Recovery

Jennifer Fichter, Alexander Bui, Greg Grawunder, and Tom Jones; Baker Hughes

Copyright 2010, AADE

This paper was prepared for presentation at the 2010 AADE Fluids Conference and Exhibition held at the Hilton Houston North, Houston, Texas, April 6-7, 2010. This conference was sponsored by the Houston Chapter of the American Association of Drilling Engineers. The information presented in this paper does not reflect any position, claim or endorsement made or implied by the American Association of Drilling Engineers, their officers or members. Questions concerning the content of this paper should be directed to the individuals listed as authors of this work.

Abstract

Deep matrix hydraulic fracturing is a precondition for transforming low permeability shale gas reservoirs into commercial assets; however, stimulating production involves more than increasing fracture permeability hydraulically. Planning and coordinating multiple services and designing multi-functional frac fluids are critical elements for project success.

Integrating the various frac services into a seamless operation requires up-front planning that includes a project survey and evaluation to determine the appropriate service and chemical options required for a low-risk, safe and productive operation.

An integral part of the planning process is the selection of frac fluid components to control bacterial growth, corrosion and scale production. An all-purpose lubricant and surface tension reducer are key components for reducing hydraulic friction and increasing flow-back volumes, respectively. Finally it is important to ensure that all the frac fluid components are compatible with each other, the frac water itself and the formation material to avoid issues during the fracturing process, flow-back period and production cycles of the well. Furthermore, an integrated chemical program from the fracturing through production will ensure a seamless transition and comprehensive risk management program throughout the life of the well.

This paper describes the process, from start to finish, how project management, careful frac-fluid additive selection and performance monitoring can optimize hydraulic fracturing operations. In addition, laboratory data are presented to illustrate the basis of fluid design and field data are presented to highlight the success of this multi-disciplined approach to improve unconventional shale gas recovery.

Introduction

Traditionally, conventional natural gas has been produced from sandstone and carbonate rock formations. More recently, however, the operators have begun to focus on unconventional natural gas reserves extracted from low permeability, tight sandstones, shale gas and coal bed methane formations to increase the production of clean burning fuel. Hydraulic fracturing is a proven technological advancement that allows natural gas producers to safely recover natural gas from deep shale formations.

The use of deep matrix hydraulic fracturing as the preferred completion technique has been a key factor in unlocking the potential of unconventional gas plays. Much has been learned since the first commercial fracture treatment was performed in the late 1940s¹. It didn't take long to discover that fractures created by hydraulic fracturing fluids tended to close off unless a proppant was included in the frac fluid design. It was also discovered that frac fluids required elevated viscosity to create adequate fracture width and proppant transport and to minimize leak-off¹.

The acceleration in gas production technology and improved hydraulic fracturing techniques can be attributed to the Barnett shale activity in an area around Fort Worth, Texas. The first Barnett horizontal well was drilled in 1992, but in the ensuing two decades sophisticated processes using horizontal drilling and sequenced, multi-stage hydraulic fracturing technologies were developed. As the Barnett Shale play has matured, natural gas producers laid the foundation for the water frac technology to spread to the other shale gas formations across the U.S. (**Figure 1**) and Canada².

The driving factors for this phenomenon were primarily tied to cost cutting, depletion of permeability or fractures that were not performing as well as expected. Aside from an increase in natural gas pricing, advances in horizontal drilling and hydraulic fracturing technology are responsible for today's unconventional natural gas recovery³.

Hydraulic Fracturing Fluids

Hydraulic fracturing involves pumping specialized fluids into a formation at a specified rate and pressure to generate fractures in the formation. For shale gas, fracture fluids are mixed with additives that help the water to carry sand proppant into the fractures. Once the fracture has initiated, additional fluids are pumped into the wellbore to increase the fracture length and to carry the proppant deeper into the formation. Additional fluid volumes are needed to accommodate the increasing length of opened fractures in the formation.

The frac fluids used for gas shale stimulations include a variety of chemical additives depending on the specific well being fractured. These chemical treatments are injected at very low concentrations with up to 12 chemical additives depending on the properties of the water and the shale formation. Each component serves a specific, engineered

purpose⁴. The predominant chemicals currently used for fracture treatments in the gas shale plays are friction-reducing additives (called slick water)⁵.

The addition of friction reducers allows fracturing fluids and proppant to be pumped to the target zone at a higher rate and reduced pressure than if water alone were used. In addition to friction reducers, other possible additives include biocides to prevent microorganism growth; scale inhibitors to prevent deposition of scale due to mixing of the fracture and connate water; oxygen scavengers and other stabilizers to prevent corrosion of metal pipe; and acids that are used to remove drilling mud damage within the near-wellbore area⁶.

Project Management

As an operator begins a new shale play fracturing program, it is critical to have a comprehensive viewpoint, not limiting the focus to the pumping of the frac job, but also considering how the drilling and stimulation processes will impact the day-to-day operations of the field. As part of this process, it is important to take a systematic approach when evaluating what chemicals/additives should be introduced to the fluids during the fracturing process. This approach involves several key process steps: 1) a detailed survey to understand the system; 2) thorough chemical selection process including both field and laboratory evaluations; 3) careful consideration on how to implement the chemical programs; and 4) a comprehensive monitoring and optimization program. The components of this process will be demonstrated in the remaining sections of this paper as we address bacterial and scale control, reduction of friction during the fracturing process and addition of Flow-Stimulator Additives to reduce the formation surface tension, allowing for faster return of the frac water.

Frac Fluid Additive Selection

Bacterial Control

Due to the large volumes of water used during the hydraulic fracturing process, the fracturing water sources are most commonly stored in lined or unlined earthen pits that are open to the atmosphere. Because the pits are open to the atmosphere, dust, rain, and surface run-off can be introduced into the pit water. The untreated fracturing waters can sit dormant in the pit for days to months prior to the start of the fracturing job. In many cases, the flowback water from the fracturing operation is reused, resulting in the mixing of several different water sources. In addition, common frac fluid additives such as polyacrylamide friction reducers, sugar-based polymers/gels and other organic compounds can serve as food sources for bacteria in the frac water. All of these practices lead to the potential for bacterial contamination in the reservoir and downhole.

If the frac fluid was not properly treated with a microbiocide to control bacterial activity, fracturing water bacteria can become established downhole and near wellbore during the fracturing process and the subsequent shut-in period⁷. Once bacteria become established downhole, the contamination can be introduced into the separator, water tanks, flow lines and disposal facilities downstream. Bacterial

contamination can result in biogenic sulfide production (souring), iron sulfide (black water) formation, plugging issues, and corrosion failures of downhole equipment, surface separation and storage tanks and flowlines. Prevention of bacterial contamination requires a quality bacterial control program.

Lubricity (Friction Reduction)

With the growing popularity of slickwater fracturing, much greater emphasis has been placed on the performance and versatility of friction reducers. Most friction reducers used are polyacrylamide-based, and can carry either an anionic, nonionic or cationic charge⁸. In most applications, anionic friction reducers are preferred due to their performance and cost relative to cationic friction reducers. As the salinity of the frac water increases, cationic friction reducers can become more economical, but usually only in water containing greater than 5% total dissolved solids.

The factors affecting friction reducer performance include pH, temperature, salinity and compatibility with other frac additives. The characteristics of friction reducers that determine performance include molecular weight, charge, unwinding/hydration speed and shelf life. Due to past problems of incompatibility-related pressure problems, all frac fluid additives must be pre-screened for compatibility and performance in source waters prior to the fracturing process. Additionally, the speed of hydration of a friction reducer polymer is critical and should be evaluated. Due to growing restrictions by regulatory agencies, greater volumes of early flow-back waters will be reused. The increasing suspended solids and salinity of frac waters will require salinity-tolerant friction reducers, such as high-brine anionic friction reducers and cationic friction reducers.

Interactions between a friction reducer and biocide can result in consumption of both products, resulting in greater quantities being needed for effective friction reduction and biocidal activity⁹. This problem is especially prevalent when the frac fluid additives are provided by more than one chemical supplier. Through laboratory and field evaluations, these interactions can be evaluated and compatibility indices can be established.

Scale Inhibition

Preventing mineral scale deposition during and after completion of the fracturing process is crucial to ensuring optimal production and longevity of the well. The deposition of mineral scale in the formation, perforations, wellbore and surface equipment can be prevented through the use of scale inhibitors applied during the fracturing process. The application regime and loading rates of scale inhibitor are dependent on variations in produced water chemistry due to geological formation diversity.

In more developed shale plays, such as the Barnett Shale, with the use of geostatistical analysis tools, operators can understand the potential scaling risk of a given well before drilling commences. **Figure 2** shows a map that illustrates the

scaling risks for barium sulfate and strontium sulfate in Barnett Shale wells. From the information gathered, presumptions can be made regarding scaling risks in a particular location, and scale inhibitor rates can be adjusted accordingly to ensure that the appropriate amount and type of scale inhibitor is present during the fracturing process and the flowback period before production scale inhibitor application begins.

Flow-Stimulator Additive

Surfactants are used in the frac fluid to lower capillary forces to assist in the recovery of the injected fluid during the production phase. Without proper screening of these molecules, surfactants can be selected that adsorb on to the fracture surfaces and cause phase trapping. The net effect of phase trapping is reduced fracture permeability and reduced production.

Laboratory testing has shown that microemulsion blends of surfactants increase frac fluid flow-back in tight shale gas reservoirs and lowers the pressure required for flowback¹⁰. Another one of the important characteristics of these fluids is their low interfacial tension¹¹. Low interfacial tension is critical because these interfacial forces are maintained as the frac fluid enters the fracture spaces and when flowback begins, the inherent mobility of the fluid is high. **Figure 3** illustrates the low interfacial tension properties of a blend of two dissimilar fluids, a microemulsion fluid and a heavy crude oil.

Another necessary characteristic of the frac fluid design is that it should lower surface tension properties between the shale surface and the produced gas. The treatment levels of this additive are low due to costs and thus, must exhibit low surface tension properties in the ppm range. **Figure 4** is a graph of measured surface tension values at various low ppm levels. Note that even at 10 ppm of active product, the surface tension is similar to the surface tension measured at higher ppm (e.g. 500, 200, 100, etc.).

Laboratory Studies

Bacterial Control

To determine the optimum biocide program for treating the fracturing fluid bacterial populations, planktonic bacterial kill studies were performed on several different chemically free frac water sources¹². The test involved inoculation of the water with previously cultured indigenous bacteria, weighing out the water into clean 6-oz glass prescription bottles, and dosing with biocides at various concentrations. In addition, a control sample with only indigenous bacteria was prepared. The analysis exposed the bacteria in each sample to the biocides for various contact times, such as 1 hour, 24 hours, 1 week and 3 weeks. The longer contact times simulated the fracturing fluid water that is retained by the reservoir following the flowback period.

At each contact time period, the serial dilution technique was used to enumerate the surviving bacteria in each biocide-treated and control sample. The acid-producing bacteria

(APB) enumeration used samples diluted into a freshwater phenol red dextrose medium, while the sulfate-reducing bacteria (SRB) enumeration used samples diluted into a freshwater proprietary SRB medium. To simulate downhole conditions, the serially diluted culture vials were incubated for 28 days at 95° F. A six-vial serial dilution was inoculated for the biocide-treated samples and an eight-bottle serial dilution series was used for the control samples.

Following the 28-day incubation period, the results of the kill study were tabulated and the 1-hour and 24-hour contact time results are reported in **Figure 4**. The results showed that Biocide A at 75 ppm and Biocide C at 150 ppm provided an eight-log reduction in both the APB and SRB concentrations as compared to the untreated control. Upon consideration of product price, Biocide A at 75 ppm was deemed to be the most cost-effective bacterial control program for this frac water source.

Lubricity (Friction Reduction)

Comprehensive laboratory evaluations of friction reducers will evaluate the effect of pH, temperature, salinity and other frac additives on the drag-reduction capabilities and dispersion speed. The temperature, salinity and pH tolerance range of a friction reducer can be established through the use of a dynamic flow loop apparatus as described by P. Kaufmen et al.⁸. **Figure 5** illustrates the brine tolerance range of an anionic friction reducer.

In order to eliminate incompatibility-related pressure risks, extensive laboratory evaluations of product compatibility must be carried out with the use of flow loop studies (**Figure 6**), biocide kill studies and scale inhibition tests. By evaluating the performance of each additive in the presence of the other additives, it is possible to quantify any potential negative or positive interactions.

Scale Inhibition

Laboratory evaluations of frac scale inhibitors require rigorous replication of the dynamic conditions both in the early stages of the fracturing process and the late stages of the flow-back. Dynamic scale tube block tests allow for accurate analysis of scale inhibitors under system pressure and temperature, providing results that are not possible with static tests¹³. **Figure 7** demonstrates the establishment of a minimum effective concentration (MEC) of a frac scale inhibitor using the dynamic scale tube block method under system conditions. By using geostatistical software in combination with the MEC results, the optimal loading rate can be established to provide seamless scale inhibition for all phases of completion and production.

Case Histories

Bacterial Control

A Barnett Shale operator was experiencing a high number of microbially induced corrosion failures in their gathering system flowlines and biogenic hydrogen sulfide gas production in their production wells and produced water storage tanks. These bacterially associated issues created a

risk of negative environmental impact and potential for personal injury. A detailed microbiological survey of the fracturing process, the gas/fluid separation facilities and the gathering system was performed to identify the source of these issues. Results for a representative wellsite survey are shown in Figure 5. The survey concluded that the source well water used for fracturing was contaminated with high levels of acid-producing and sulfate-reducing bacteria (typically 10^4 to $>10^6$ APB and SRB/mL (**Figures 8, 9 and 10**).

The incumbent microbiocide program for the frac water was ineffective, resulting in contamination of the production wells during the frac job and subsequent contamination of the downstream portions of the system as the fracturing fluid was flown back and the well was put on production. The representative planktonic kill study on the frac water (**Figure 11**) indicated that Biocide A at 50 to 75 ppm would be the most cost-effective biocide for treating the frac water. The biocide was injected at 60 ppm “on the fly” into the blender with a dedicated frac chemical injection truck.

Monitoring frac water samples were collected just prior to pumping the frac job to determine the background concentration of bacteria. Following the frac jobs, additional samples were collected from the production wellhead to determine the surviving concentration of bacteria. Samples were collected within 10 days following the frac job (early flowback), 30 days, 60 days and 90 days post-frac. A target bacterial concentration of $\leq 10^3$ bacteria/mL was set as the performance target for the biocide program. The early flowback results for 70 wells treated with 60 ppm Biocide A demonstrated that 95% of the wells treated had bacterial concentrations within the target specification (**Figure 11**).

Friction Reduction

An operator producing in the Barnett Shale had been experiencing pressure problems during frac jobs leading to higher horsepower requirements, longer pumping times and added expense. The issue traced back to frac additive incompatibility issues between the friction reducer and other crucial additives. Because these operators were sourcing additives both from the frac pumping company and a chemical service provider, there was no effective way to predict or address chemical incompatibilities. The operator sought a single supplier that could provide a complete range of high-performing and compatible additives in order to reduce costs and bring wells online sooner.

Fit-for-purpose product and service recommendations were provided based on water chemistry, measured bacterial populations and reservoir pressures. Full laboratory support was deployed to ensure product compatibility before any chemical was applied. Through careful product selection and application optimization, the operator enjoyed a 5 to 10% reduction in friction reducer injection rates relative to prior frac jobs (often getting effective results at rates less than 0.25 gallons per thousand, **Figure 12**) Biocide injection and scale inhibitor rates were also optimized resulting in significant cost savings to the operator. Most importantly, compatibility testing ensured that neither the biocide nor the scale inhibitor

retarded the performance of the friction reducer (**Figure 13**). As a result, the operator was able to safely overcome and stabilize reservoir pressure spikes and maintain high rates of injection.

Scale Inhibition

An operator in the Barnett Shale was experiencing increasing reports of plugged or restricted tubing within the first 30 days of production. Laboratory analysis of the solids indicated deposition of barium sulfate and strontium sulfate. Through careful monitoring of produced water after the fracturing process, it is possible to determine the effectiveness of a scale inhibition program. **Figure 14** demonstrates the scaling tendencies typically experienced in the Barnett Shale over the first five months. As seen in the graph, the sulfate that was introduced via the frac source water declined rapidly, but the increasing salinity and barium in the produced water created a significant scaling potential for barium sulfate while the sulfate was still in the well, which was 15 days post-frac for this well. From this example, there was enough scale inhibitor present above its minimum effective concentration to prevent barium sulfate formation. Through the use of the geostatistical predictive tools and laboratory analyses, costly mineral scale deposition can be prevented.

Flow-Stimulator Additive

An operator in East Texas completed several hydraulically fractured wells located within a half-mile radius. One well was treated with a Flow-Stimulator Additive during the fracturing process and compared with wells that were not. From the operator’s perspective, the Flow-Stimulator Additive has significantly increased the total production volume, by 144% and 141%, based on 30 and 60 days of production, respectively. Because of its ability to improve water flow-back, solubilize emulsions and sustain total production, several hundred of barrels of incremental oil were also realized during the first 60 days of production.

Conclusions

Hydraulic fracturing using slick water is a common mechanism to convert low permeability shale gas reservoirs into commercial assets. During the planning stages of the fracturing process, it is important for operators to think long-term and consider the impact the fracturing process might have on the day-to-day operations once the wells have been brought on production. Planning and coordinating services and designing multi-functional frac fluids are critical elements for project success.

Critical to determining the essential frac fluid additives is an up-front project survey and system evaluation to anticipate the operational issues that may arise due to the fracturing process and determine the appropriate service and chemical options required for a low-risk, safe and productive fracturing operation.

Once the field survey is complete and the fracturing process and system operations are well understood, another essential step in designing a fit-for-purpose frac additive program for an operator is to perform detailed laboratory

evaluations for product selection, mimicking system conditions as closely as possible. For scale inhibitor product selection, dynamic scale tube block tests allow for rigorous replication of the dynamic conditions occurring in the early stages of the fracturing process and the late stages of the flow-back, allowing for duplication of the system pressure and temperature. Flow loop testing under system conditions will allow for determination of the optimum friction reducer chemistry and loading rate for reducing hydraulic friction. Laboratory testing allows for determination of the optimum microemulsion surfactant blends for increasing fracture fluid flow-back in tight shale gas reservoirs and lowering the pressure required for flow-back. Planktonic bacterial kill studies should be performed using representative system waters for selection of the most cost-effective bacterial control program. Once all the frac additives and loading rates have been determined, it is imperative to ensure that all the frac fluid components are compatible with each other, the frac water itself, the production chemicals and the formation material to avoid issues during the fracturing process, flow-back period and production cycles of the well.

An aggressive monitoring program is instrumental in assessing the performance of the frac chemical program. However, collection of the data is not enough. It is so important to take the time to learn from the information gained from the monitoring program and use the data to optimize the chemical program and assess system conditions that would require an adjustment of the chemical loading rate.

Finally, an integrated chemical program from the fracturing through production will ensure a seamless transition and comprehensive risk management program throughout the life of the well.

References

1. Howard, G.C. and C.R. Fast (editors), "Hydraulic Fracturing, Monograph Vol. 2 of the Henry L. Doherty Series," SPE 027, New York, 1970.
2. Hayden, J., and D. Pursell, D. *Pickering Energy Partners Inc. The Barnett Shale*. Visitors Guide to the Hottest Gas Play in the US, <http://www.tudorpickering.com/>, /, October 2005.
3. Energy Information Administration, Is U.S. Natural Gas Production Increasing? Energy in Brief, June 2008.
4. EPA. Drinking Water Academy (DWA). Introduction to the Underground Injection Control Program, January 2003.
5. EPA. US EPA's Program to Regulate the Placement of Waste Water and other Fluids Underground. EPA 816-F-04-040, June 2004.
6. Schlumberger Fracturing Services PowerSTIM, www.slb.com, September, 2008.
7. J. Fichter, K. Johnson, K. French, R. Oden. "Use of Microbiocides in Barnett Shale Gas Well Fracturing Fluids to Control Bacterially Related Problems," NACE 1703, NACE Corrosion New Orleans, LA., March 16-20, 2008.
8. P. Kaufman, G.S. Penny, and J. Paktinat., "Critical Evaluations of Additives Used in Shale Slickwater Fracs," SPE 119900, SPE Shale Gas Production Conference, Fort Worth, TX, 16-18 November, 2008.
9. S.M. Rimassa, P.R. Howard, M.O. Arnold. "Are You Buying Too Much Friction Reducer Because of Your Biocide?" SPE 119569-MS, SPE Hydraulic Fracturing Conference, The Woodlands, TX, January 19-21, 2009.
10. J. Paktinat, A. Pinkhouse, N. Johnson, C. Williams, G. Lash, G. Penny and D. Goff. "Case Study: Optimizing Hydraulic Fracturing Performance in Northeastern United States Fractured Shale Formations," SPE 104306, SPE Eastern Regional Meeting, Canton, Ohio, 11-13 October, 2006.
11. R. Peresich, T. Jones, L. Quintero, T. Gardin. "Case Studies of Mesophase Technology Employed for the Remediation of Case Hole Completions," NTCE 18-01, AADE Annual Conference and Exhibition, New Orleans, LA., 2009.
12. NACE *Standard TM- 0194-04* Field Monitoring of Bacterial Growth in Oilfield Systems, 2004.
13. M.D. Yuan, E. Jamieson, P. Hammong, Baker Petrolite, Investigation of Scaling and Inhibition Mechanisms and the Influencing Factors in Static and Dynamic Inhibition Tests

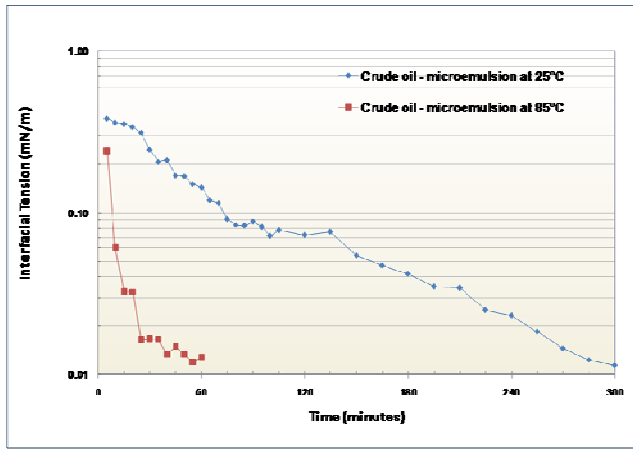


Figure 4 IFT of flow-stimulator additive in crude oil

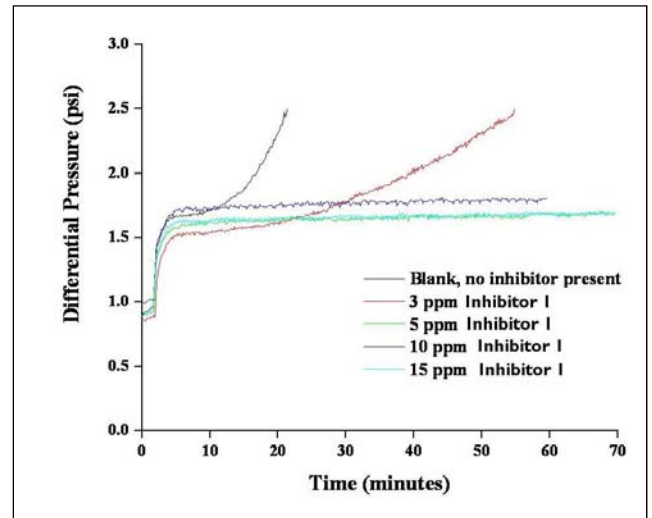


Figure 7 Dynamic scale tube block testing of scale inhibitor

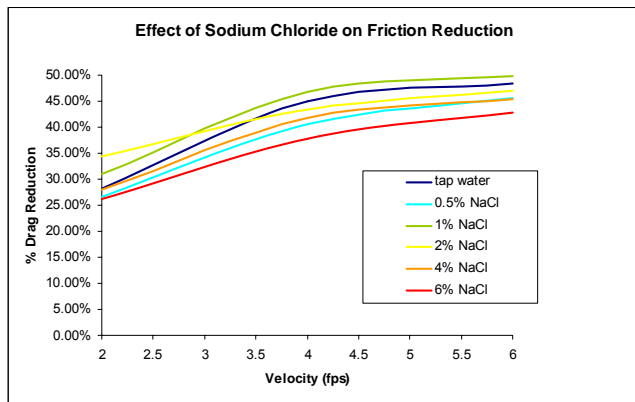


Figure 5 Effect of salinity on anionic friction reducer

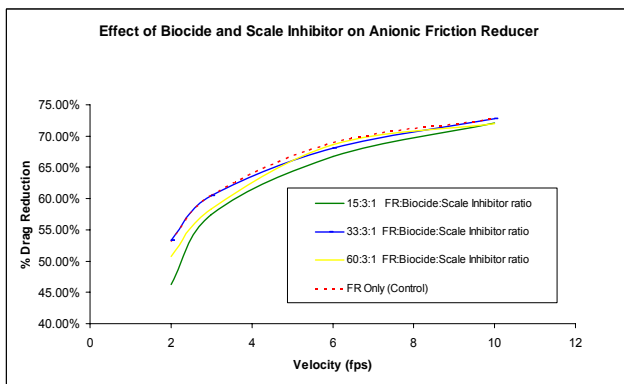


Figure 6 Effect of biocide and scale inhibitor on anionic friction reducer

Sample Location	Microscopy (Bacteria/mL)	Bacterial Culture Media	
		APB/mL	SRB/mL
Source Well	2 X 10 ⁵	10 ⁵	BD
Frac Pit	7 X 10 ⁶	≥ 10 ⁶	10 ²
Wellhead	3 X 10 ⁵	10 ⁴	10 ²
Separator	2 X 10 ⁶	≥ 10 ⁶	10 ³
Produced Water Storage Tank	2 X 10 ⁷	≥ 10 ⁶	≥ 10 ⁶

BD = bacterial concentrations are below the detection limit of the assay (< 1 bacteria/mL)

Figure 8 Microbiological survey results for a representative wellsite

Sample ID	Microscopic Analysis		Bacterial Culture Media		Water Quality
	Bacteria/mL	Algae/mL	APB/mL	SRB/mL	
Pond #1	5 X 10 ⁶	occasional	≥ 10 ⁶	10 ⁴	Tan water with solids; natural stock pond
Lined Pit #1	9 X 10 ⁶	BD	≥ 10 ⁶	10 ³	Opaque water
Pond #2	2 X 10 ⁶	3.4 X 10 ⁴	≥ 10 ⁶	10 ⁴	Opaque water
Pond #3	4 X 10 ⁵	occasional	10 ⁵	10 ²	Opaque water
Lined Pit #2	9 X 10 ⁷	BD	≥ 10 ⁶	≥ 10 ⁶	Black water; lot of sediment
Lined Pit #3	3 X 10 ⁵	BD	10 ⁵	10 ²	Clear water
Lined Pit #4	3 X 10 ⁷	BD	≥ 10 ⁶	≥ 10 ⁶	Dark brown water

BD = below detectable limits

Figure 9 Microbiological survey results for representative fracturing water sources

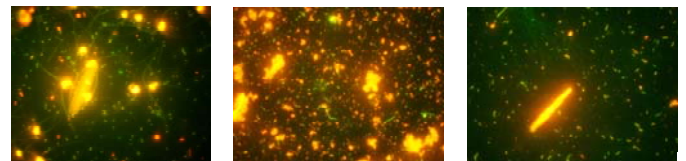


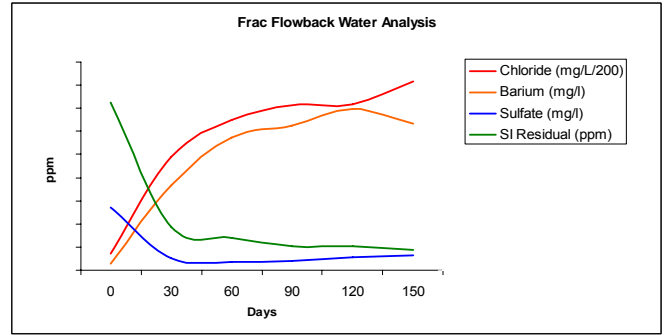
Figure 10 Photomicrographs of representative fracturing water sources

Biocide	Concentration (ppm)	One Hour Contact Time		24 Hours Contact Time	
		APB	SRB	APB	SRB
Biocide A	30	10 ⁵	10 ⁵	10 ⁵	BD
	50	10 ³	BD	10 ²	BD
	75	BD	BD	BD	BD
	100	BD	BD	BD	BD
Biocide B	50	≥ 10 ⁶	≥ 10 ⁶	≥ 10 ⁵	≥ 10 ⁵
	100	≥ 10 ⁶	10 ⁴	10 ⁵	≥ 10 ⁶
	150	10 ⁵	10 ³	10 ⁴	10 ⁴
Biocide C	200	10 ²	10 ²	10 ²	10 ²
	50	10 ⁵	10 ⁵	10 ⁵	10 ²
	100	10 ²	BD	10 ³	BD
Biocide D	150	BD	BD	BD	BD
	200	BD	BD	BD	BD
	30	≥ 10 ⁶	≥ 10 ⁶	≥ 10 ⁶	≥ 10 ⁶
Biocide E	50	≥ 10 ⁶	≥ 10 ⁶	≥ 10 ⁶	≥ 10 ⁶
	75	≥ 10 ⁶	≥ 10 ⁶	≥ 10 ⁶	≥ 10 ⁶
	100	≥ 10 ⁶	≥ 10 ⁶	≥ 10 ⁶	≥ 10 ⁶
Biocide F	25	≥ 10 ⁶	≥ 10 ⁶	≥ 10 ⁶	≥ 10 ⁶
	50	≥ 10 ⁶	≥ 10 ⁶	≥ 10 ⁶	≥ 10 ⁶
	100	≥ 10 ⁶	≥ 10 ⁶	10 ⁵	10 ³
Untreated Control	---	10 ⁶	10 ⁶	10 ⁶	10 ⁶

*Results are expressed as number of positive bottles in a serial dilution series; a 9 bottle series was inoculated for the untreated control; a 6 bottle series was inoculated for all treated samples.

≥ 10⁶ = all 6 bottles in the serial dilution series were positive.

Figure 11 Representative planktonic kill study results



Day	BaSO4 Saturation Index	Predicted BaSO4 Amount (lbs/1000bbls)
0	2.3	78.2
15	2.67	254.1
30	2.51	172.1
45	2.56	124.1

Figure 14 Scaling tendencies in first five months of production

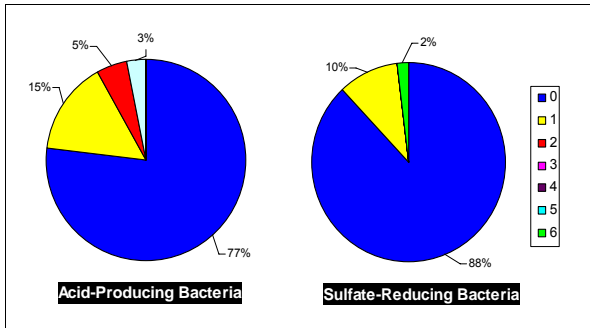


Figure 12 Flowback monitoring results for 70 production wells where fracturing fluid was treated with 60 ppm Biocide A. Results expressed as number of positive culture media bottles in a serial dilution series.

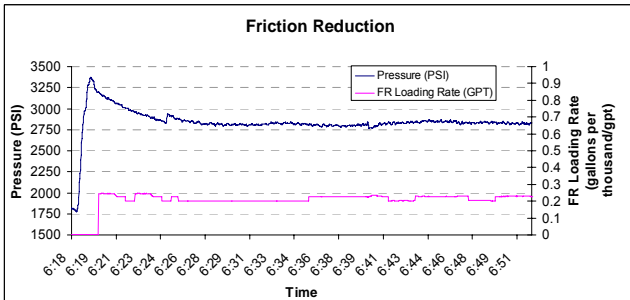


Figure 13 Friction reductions during fracturing process