SPE 141211

Case Study: Evaluation of an Oxidative Biocide During and After a Hydraulic Fracturing Job in the Marcellus Shale

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This paper was prepared for presentation at the SPE International Symposium on Oilfield Chemistry held in The Woodlands, Texas, USA, 11–13 April 2011.

Abstract

Effective microbiological control is an important aspect of a successfully executed fracturing job. Control of bacterial growth is often accomplished through the use of biocides such as glutaraldehyde, particularly in the multi-stage, high-volume fracturing of unconventional shale gas reservoirs. Biocidal additives, which are toxic by necessity, can persist in flowback water, so their use in shale fracturing has come under increasing scrutiny since high biocide concentrations in flowback water increase fluid cost and limit the options for disposal. The case for designing a bactericide program to match, and not exceed, the required amount of bacterial control is clear, but rarely is the bacterial load determined during and after the job to verify this balance.

Herein, we report a case study undertaken to evaluate the bacterial load of field mix water and flowback water during and after a large hydraulic fracturing job in the Marcellus Shale. A novel oxidative biocide product was used during the fracturing job that has both an effective fast kill and a low toxicity profile (e.g. HMIS rating of 1,0,0). Because of its rapid biodegradability, there was concern that the effective kill of this biocide would not persist beyond a few days. Industry standard techniques (NACE Std. TMO194-94) for quantifying bacteria were applied to water samples taken during the job and over several weeks of production. The biocide was also evaluated for compatibility with common fracturing additives and for its corrosivity to surface equipment and tubular goods.

This study determines that the new biocide does not persist in flowback water beyond a few days. However, analysis of flowback water samples reveals that the bacteria count stays low (less than 10 cells/mL) for up to 81 days after application of this biocide in a slickwater fluid. Additionally, genetic fingerprinting using Denaturing Gradient Gel Electrophoresis Analysis (DGGE) was applied to the bacteria in the initial field mix water to allow comparison to any bacteria detected in the flowback samples. This paper will describe the details of this case study.

Since the completion of this case study, we have successfully deployed this technology on treatments in the Barnett, Haynesville, Marcellus, and Granite Wash shale regions. This paper reveals details of a field test and of the efficacy of this biocide as tested in flowback waters from the Piceance and Marcellus Shale basin. The results of the bacteria enumerated from each job site sample are presented. Finally, dosage requirements for biocidal efficacy were optimized for slickwater hydraulic fracturing applications are described.

Introduction

Control of microbial growth is an essential consideration in the design of fracturing fluids. (Brandon, et al., 1995) Because of their ability to rapidly degrade biopolymers such as guar, bacterial enzymes can seriously affect the rheology of traditional gels. Slickwater fracturing fluids, where viscosity is not critical and the (typically synthetic) drag-reducing polymers are unaffected by bacterial enzymes, also require a biocide strategy to prevent well damage. The re-use of produced water (PW) in slickwater campaigns raises the risk of introducing anaerobic bacteria to the well, because PW is generally less oxygenated (Seright, et al., 2009) and is often rich with inorganic nutrients. Acid-producing bacteria (APB), and sulfate-reducing bacteria (SRB) can cause problematic localized corrosion to completions and tubular goods. (Nemati and Voordouw, 2000) The latter can also be responsible for well souring and iron sulfide precipitation in the low-oxygen wellbore environment. (Carpenter and Nalepa, 2005) These and other general heterotrophic bacteria (GHB) can form biofilms that damage proppant packs and impair production flow, and can be problematic for surface equipment and even pipelines. (Bottero, et al., 2010).

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It is far easier to plan to kill or control bacteria in the fluid than it is to remediate these problems once they are detected in the producing reservoir. In the slickwater fracturing context, this means killing or controlling bacteria in the mix water (the primary source of bacteria) and offering some residual control of bacteria in the fluid to deal with adventitious microbes that might be incorporated during blending. Bacteria that are introduced at surface (e.g. with sand or in mix water drawn from various surface sources) will generally be mesophilic aerobes, dependent on oxygen and not able to survive for long above 125 °F (~50 °C). (Talaro and Talaro, 1999) Anaerobic APB, SRB and GHB in recycled PW will likely be dormant in water oxygenated by aeration during blending or by extended time in surface containment (e.g. ponds, tanks), but they can flourish downhole if re-injected. Clearly, effective initial control of mix water bacteria coupled to some residual downhole biocidal activity represents a good biocidal strategy for slickwater fluid design.

Successful chemical control methods can be separated into two broad classes based on mechanism of action: (1) non-oxidizing (sometimes called 'preservative'), and (2) oxidizing. Historically, the non-oxidizing biocides are the antimicrobial chemicals most often used in hydraulic fracturing fluids. Typically, the two classes have roughly the same biocidal effectiveness, (Maillard, 2002) but fast-acting oxidizing biocides can react chemically with other fluid components and the fluid environment, where slower-acting non-oxidizing biocides can interact minimally with other additives if they are selected carefully. (Rimassa et al., 2008) Non-oxidizing biocides do not spend themselves quickly in the wellbore environment. As a consequence, non-oxidizing biocides can persist in returned fluids longer than is necessary. Since non-oxidizing biocides are intrinsically hazardous and often toxic, they have unique wellsite handling and HSE issues. High biocide concentration in flowback water limits disposal options and increases handling costs. Persistent non-oxidizing biocides are not desirable in groundwater or in aquifers.

In contrast, the bactericidal action of oxidizing biocides is faster than that of non-oxidizing biocides. (Talaro and Talaro, 1999; Maillard, 2002) There is a commonly-held belief that this speed implies that oxidizing biocides do not have sufficient persistence to effectively suppress bacterial regrowth. The object of this study was to determine if an oxidative biocide can successfully control long-term bacterial growth in well treatments.

For any chemical additive, wellsite delivery is an important consideration. For example, oxidative chemicals such as breakers are widely understood to have an associated reactivity hazard. Non-oxidative biocides are inherently toxic and need to be handled with care. A consideration in our choice of oxidative biocide was to match a safe wellsite delivery profile (e.g. HMIS rating 1,0,0) with optimal bacterial control. This is a particular advantage in the modern slickwater fracturing market, where treatment volumes run into the millions of gallons in multiple stages per well and the public profile of chemical additives is much higher than in the past.

We have cited some reports above that illustrate the successful use of biocides in the oilfield. Few reports exist that actually evaluate the efficacy of the biocide while pumping the treatment at the wellsite. Herein, we discuss the evaluation of an oxidative biocide that is compatible with slickwater fluids, and we demonstrate its successful use in a hydraulic fracturing treatment in the Marcellus shale. Efficacy of this environmentally responsible oxidative biocide was determined while pumping the treatment and at post-treatment times up to 81 days after the treatment. The genetic material of the bacteria detected in the field mix water used in the treatment was determined by denaturing gradient gel electrophoresis (DGGE) and compared the fingerprint to the initial flowback water sampled 21 days after the treatment. Laboratory testing to determine the efficacy of this biocide with other shale basin produced waters will also be discussed.

Experimental

Materials

Water samples used in the microbiology study were obtained from a field location (NE Pennsylvania). Water samples used for laboratory testing of additive compatibilities were prepared using PW obtained from the Piceance Basin (PB-PW). An environmental consortium of GHB, APB, and SRB cultured from oilfield production systems operating at an equivalency to the PB-PW sample was used to inoculate a base culture stock of bacteria for biocide testing. The base stock culture was created to prevent toxicity and/or bacterial transfer shock from potentially affecting results and data interpretation. The newly inoculated stock cultures were then incubated at 35 ºC for two to four days to revive the bacteria and promote growth. Standards used for water analysis were obtained commercially. Standards and samples used in water analyses were diluted with deionized water.

Water analysis

Analysis of dissolved cations was performed by inductively coupled plasma atomic emission spectrometry (ICP-OES) using a PerkinElmer Optima 2000 DV using serially-diluted calibration standard solutions that included all of the metal analytes. The spectrometer uses a double monochromator optical system for high resolution and has a dual backside-illuminated charge coupled device as the detector. Field samples were diluted to 1:1000 before analysis (Error ±0.1%). Anion analysis was carried out using separate titrimetric procedures for the determination of chlorides, bicarbonates, and hydroxides (Error ±15%). The sulfate concentration was determined using a sulfate test strip (Error ±10%). Total dissolved solids (TDS) concentration was determined using a standard laboratory method (Error ±15%). (Eaton, et al., 1995)

Biocide Efficacy Tests
Bottle tests were used to enumerate the surviving planktonic and sessile bacteria population over a seven day period following NACE TMO194-94 recommendations for microbial monitoring in oilfield systems. The data was then evaluated against two controls. Denaturing Gradient Gel Electrophoresis was performed according to standard biochemical techniques. (Muyzer and Smalla, 1998; Teske, et al., 1996)

Rheology tests
All rheology tests were performed using a Chandler viscometer, Model 5550. The mixing procedure for the fracturing fluids was performed as follows: 500 mL of deionized water was placed into a Waring blending cup. Subsequently, the oxidative biocide was added and allowed to mix for 20 seconds. The gelling agent was then added and allowed to mix for 10 minutes, after which the linear gel viscosity was checked and compared to the hydration chart. The remaining additives were then added to the solution and the vortex was allowed to close after the addition of the crosslinker.

Corrosion Tests
N80 test coupons used for evaluation were measured as having the following properties: 28.2 cm^2 surface area and 1.63 × 1.0 in (4.14 × 2.54 cm) dimensions. Coupons were numbered for identification, bead-blasted to remove mill scale, and stored in a desiccator. Prior to treatment, the coupons were cleansed, rinsed in acetone, dried, and accurately weighed. Coupons were exposed to treatment fluid in an autoclave at 100 ºF at 3000 psi for eight hours. Following the test, the coupons were rinsed in acetone and scrubbed with soap and water to remove corrosion deposits. A final rinse in acetone was completed prior to re-weighing the metal coupons for calculation of corrosion rates based on mass loss. The maximum allowable corrosion rate recommended for these test conditions is 0.05 lb/ft^2 with acceptable pitting. Pitting is reported using a "pitting index, where 0 = none, 1 = minor edge corrosion, 2 = edge pits only, 3 = fewer than 25 pinpoint pits on curved surfaces, 4 = greater than 25 pinpoint pits on curved surfaces. (Smith et al., 1978) A pitting index of 2 or lower is acceptable. The strip factor and corrosion rate are calculated as follows (Equations **1** and **2**):

$$\text{Strip Factor (SF)} = \frac{\text{coupon weight}}{\text{coupon area}} \qquad \text{Eq. } \mathbf{1}$$

$$Corrosion\ Rate\ (lb/ft^2) = \frac{weight\ lost}{original\ weight} \times SF \qquad \text{Eq. } \mathbf{2}$$

Friction Loop Analysis
Friction reduction for slickwater fluids is measured using a friction loop consisting of a pump, a pipe of known dimensions and a tank connected to the closed loop. The tank feeds the pump and collects the effluent from the pipe section during pump operation. The friction loop allows measurement of the frictional pressure drop across the pipe section at different flow rates. In this study, a friction loop consisting of a ½" (1.27 cm) and a ¾" (1.91 cm) pipe was used for the drag reduction measurements. The pressure difference (ΔP) across the pipes, the mass flow and the temperature were recorded for each fluid analyzed. The friction loop was calibrated with tap water prior to any fluid testing and all tests were run at room temperature (75 °F or 24 °C).
For variable rate tests, the fluid is prepared ahead of each test by addition of the required additives to 15 liters of synthetic PW prepared according to the water analysis for PB-PW in a 5 gallon (0.019 m^3) plastic bucket agitated by a paddle mixer operating at 1000 rpm for 2 minutes prior to transfer to the loop. During the variable rate test, the test fluid is pumped for about 10 sec at incremented rates of 6 kg/min and %DR (drag reduction) is calculated using Equation **3**:

$$\%DR = \frac{\Delta P_{water} - \Delta P_{fluid}}{\Delta P_{water}} \cdot 100 \qquad \text{Eq. } \mathbf{3}$$

where ΔP_{water} = differential pressure of water in tubing; ΔP_{Fluid} = differential pressure of the fluid in tubing; and %DR = percent drag reduction with respect to water, dimensionless.

Results and Discussion

Laboratory Evaluation
Prior to using the oxidative biocide in a field application, lab tests were conducted to confirm the effectiveness of the biocide as well as its compatibility with fluid additives and stability over time.

Water Analysis
Produced water from the Piceance Basin (PB-PW) was used in all laboratory tests unless otherwise noted. This water was found to have 143,000 mg/L TDS, the total organic carbon (TOC) content was 801 mg/L and the pH was 8.06. Since the produced water sample had a high TDS and TOC value, 5 ppm of the oxidative biocide was used in the following laboratory tests unless otherwise stated.

Laboratory Microbiological Tests

Bottle tests were used to evaluate the biocidal efficacy of the oxidative biocide and the commonly used oilfield biocide glutaraldehyde against APB, SRB, and GHB. Friction reducer was included in each test to simulate treatment fluid. For all tests, the appropriate biocide was added to PB-PW and allowed to mix for five minutes prior to the addition of the friction reducer. The bacterial population was measured immediately upon mixing, at 5 minutes, at 30 minutes, at 24 hours, and finally at 7 days.

In a control experiment, the effect of the friction reducer on the bacterial population was found to be minimal. To establish a baseline for bacterial control in a typical fracturing fluid, glutaraldeyde and friction reducer (263 ppm each) were added to PB-PW. Figure 1 compares the effects of glutaraldehyde and the oxidative biocide on the bacterial population. Glutaraldehyde is a generally effective oilfield biocide, but it is not known whether the action of glutaraldehyde is affected by the salt and/or organic carbon content of PW. Figure 1a (left) shows that glutaraldehyde is mildly effective in PB-PW at this concentration. After 24 hours, bacterial population is reduced by two orders of magnitude. After seven days, regrowth of bacteria is apparent, suggesting the possibility of sour wells after fracturing treatment when glutaraldehyde is used. In contrast, Figure 1b (right) shows that the oxidative biocide provides very effective bacterial control. Within five minutes, the bacterial population is reduced by five orders of magnitude, from 10^6 cells/mL to 10^1 cells/mL. After 24 hours, the SRB population is not detectable and regrowth was not apparent. After seven days, all bacterial counts remain below detectable limits and no regrowth was apparent. The oxidizer is effective even in high TDS water with residual organic carbon, and it reduced the bacterial population effectively in 5 minutes whereas glutaraldehyde slowly decreased it over 24 hours without preventing regrowth.

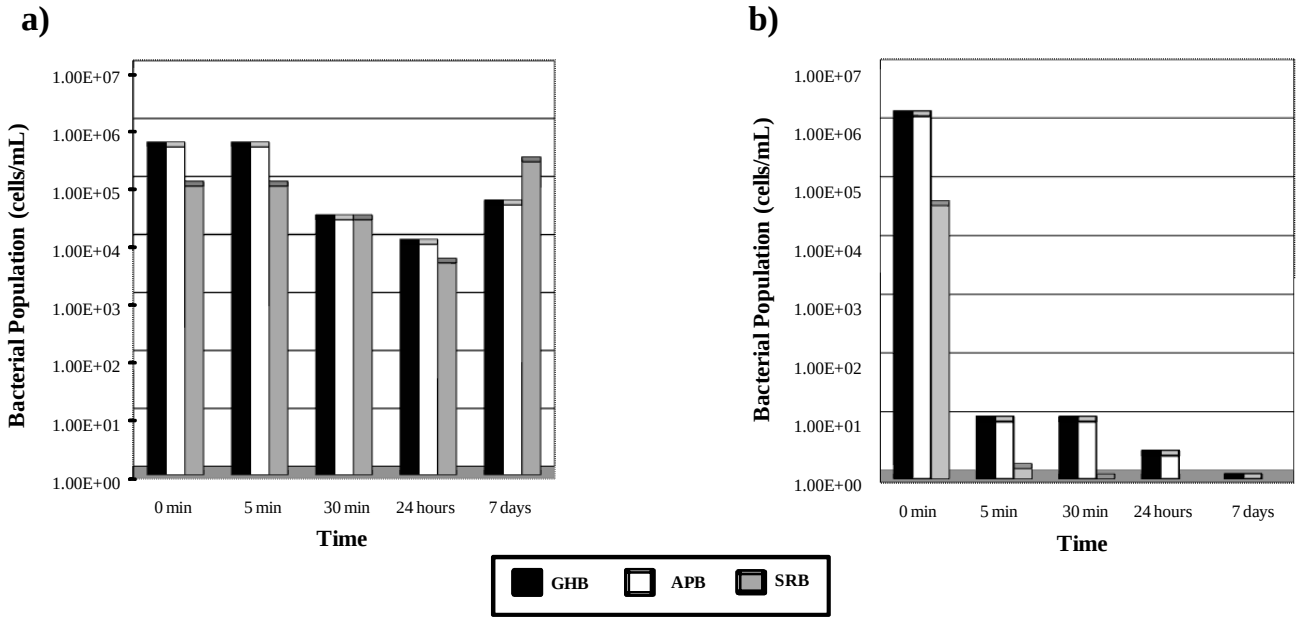

Figure 1 Laboratory bacterial test results, including a) 263 ppm glutaraldehyde in the Piceance Basin water sample and b) the 5 ppm oxidative biocide in the Piceance Basin water sample, over a seven day period. The Piceance Basin water sample was tested and had a pH value of 8.06 and a total dissolved solids concentration of 143,000 ppm.

Friction Loop tests

There is a natural concern that the oxidizing action of the candidate biocide will degrade friction reducer performance. Figure 2 shows drag reduction in PB produced water containing a multicomponent slickwater additive package with and without oxidative biocide. The data are plotted as percent drag reduction versus flow rate (kg/min). It is clear that the oxidative biocide has no effect on friction reduction.

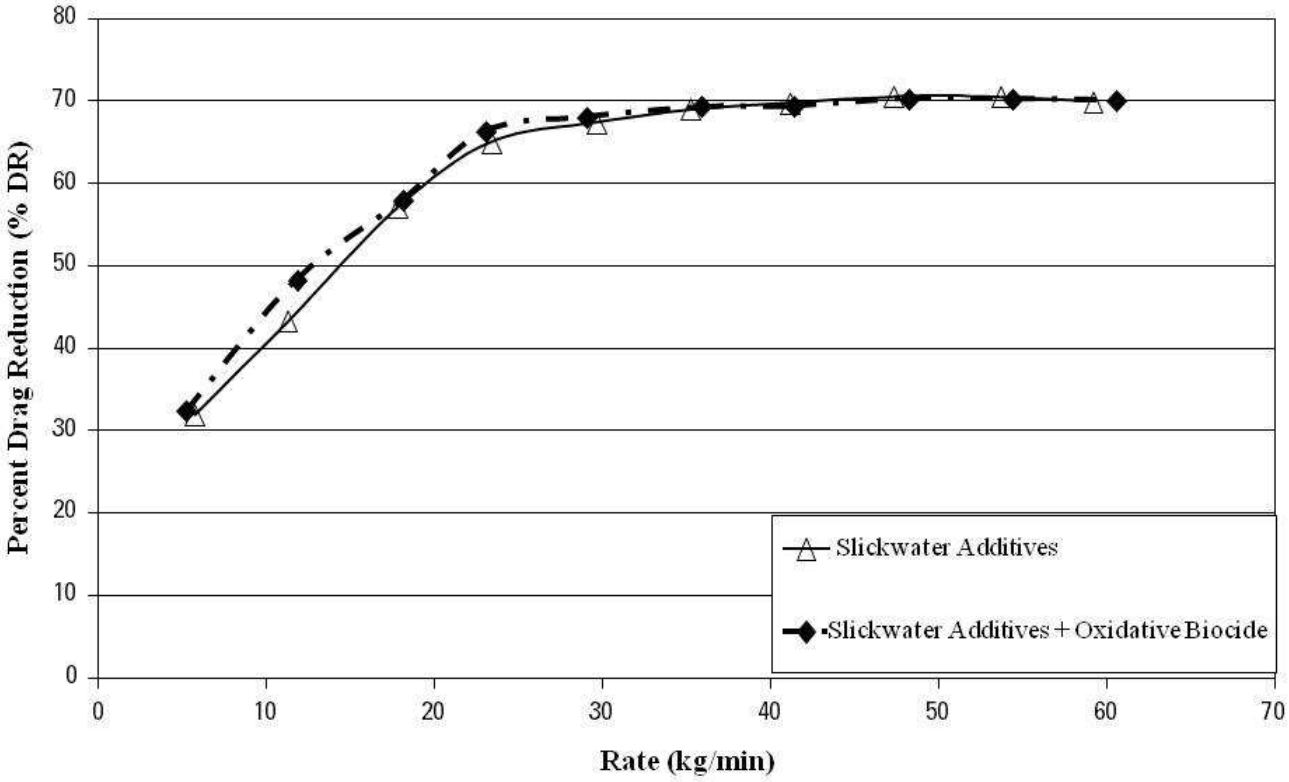

Figure 2 The maximum percent drag reduction (%DR) value for the Piceance Basin (PB) water sample containing slickwater additives (2000 ppm clay stabilizer, 275 ppm friction reducer, 109 ppm scale inhibitor, and 1818 ppm microemulsion solvent and PB water sample) and 5 ppm oxidative biocide. The Piceance Basin water sample had a pH value of 8.06 and 143,000 ppm TDS.

Rheology tests

Compatibility of the oxidative biocide with a common crosslinked guar fracturing fluid currently used in field operations was evaluated using a Fann50-type Chandler 5500 HPHT viscometer, following the API RP 39 schedule. The fluid was a 20 lb gel crosslinked at pH 11.3 to 11.4. The oxidative biocide was used at net oxidizer concentrations of 5 and 10 ppm and was added to the water at the same time as the polymer. The fluid was then tested at 150 ºF for a period of two hours. A rheology profile is shown in Figure 3. The data is plotted as viscosity, measured in centipoise (cP), as a function of time. Biocide concentration can be safely increased from 1 to 5 ppm without compromising fluid viscosity. At 10 ppm, fluid viscosity begins to be impaired. This experiment is representative of the broad compatibility of the oxidative biocide with a number of fracturing fluids.

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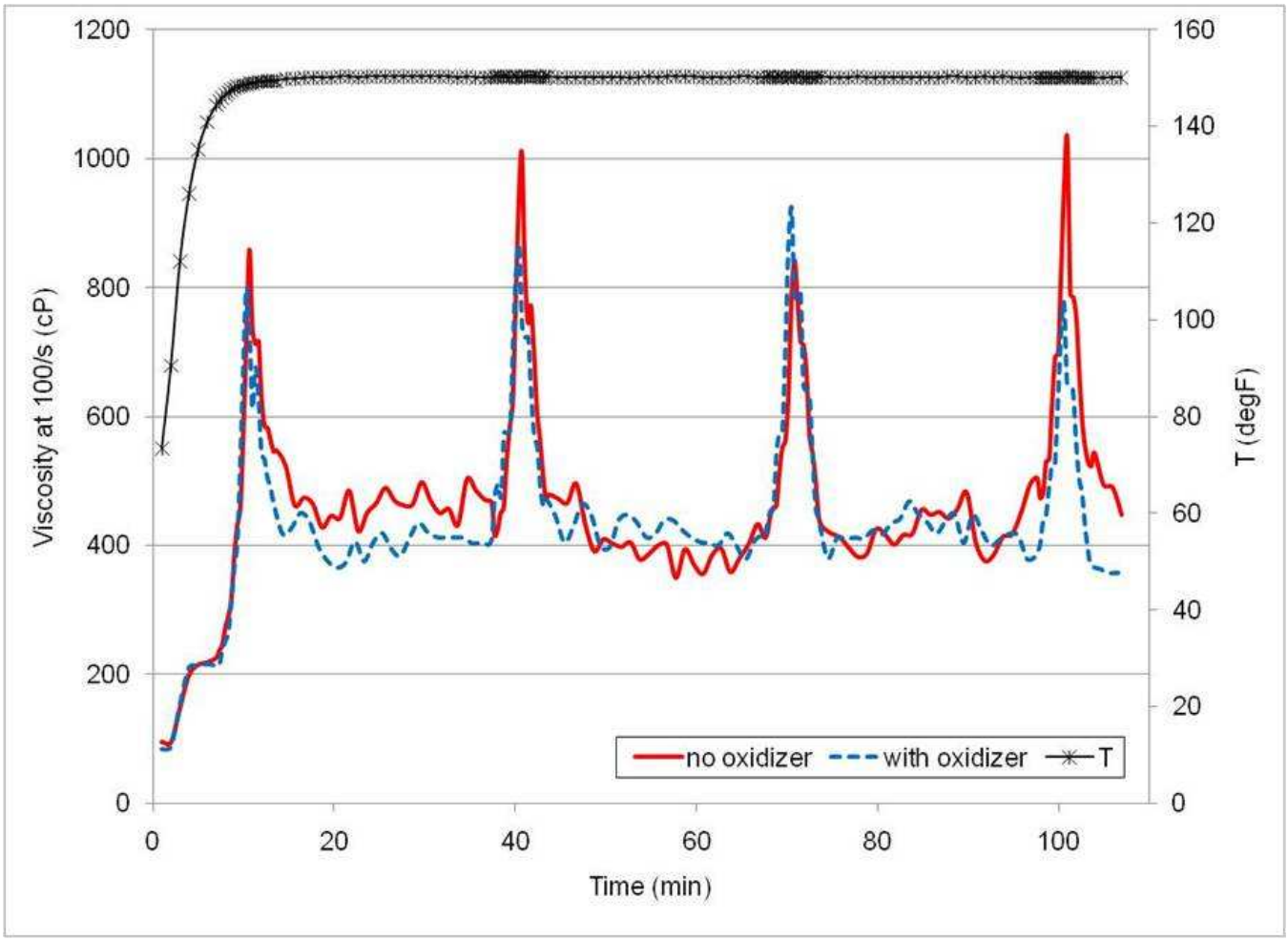

Figure 3 Rheology profile of a crosslinked 20 lb guar gel at pH 11.25 with 5 ppm oxidative biocide at 150 ºF.

It is clear from this test and from other experiments using related fluids that the oxidative biocide is generally highly compatible with standard gelled fluids in addition to slickwater fluids.

Corrosion Tests

The oxidative biocide was evaluated at various concentrations in fresh water for corrosion on N80 steel coupons at 100 ºF for eight hours at 3000 psi. All coupons tested yielded very low corrosion rates: coupons showed minimal loss of mass and no pitting was observed. The corrosion evaluation data is shown in Table 1**.** The oxidative biocide is therefore not harmful to surface equipment or oilfield tubular on the timescale of a typical slickwater treatment.

Table 1 Corrosion Test results show negligible corrosion to typical oilfield tubular goods across appropriate concentration ranges of the oxidative biocide (tests were for 8 hours at 100 °F)

Loading (ppm)	Pitting Index	Corrosion Rate (lb/ft^2)
5	0	0.0001
10	0	0.0001
20	0	0.0002
50	0	0.0003

Assured that the oxidative biocide is non-corrosive and fully compatible with slickwater and crosslinked guar fluids, we moved to field evaluation. A key wellsite objective is therefore to use the oxidative biocide in a form that is safe for transport and use.

Well Operations and Flowback Analysis

Unconventional gas reservoirs use high rate hydraulic fracturing techniques to stimulate the low permeability reservoirs. These reservoirs are typically shale formations, such as the Barnett shale in Texas or the Marcellus shale in Pennsylvania and New York. High rate hydraulic fracturing consists of pumping water, a friction reducer, support chemicals, and proppant at

high rates, up to 120 barrels per minute (bpm). The friction reducer is used to counteract the increased pressure observed when pumping at high rates. The support chemicals can include biocides, scale inhibitors, and clay stabilizers. Mix water comes from various sources including PW. The fracture design that gives effective stimulation in shale plays requires large volumes of water, which can be very difficult to supply especially in remote areas. This has led production companies to use new sources of water. For example the water that is produced from one well can be used to fracture another.

The two horizontal wells in this case study were drilled on a single pad into the Marcellus shale play. The reservoir temperature was 132 °F with measured depths starting at 11,403 ft and 11,375 ft as described in Table 2. Seventeen stages, eight on Well 1 and nine on Well 2, were pumped at 90 bpm with a clean fluid stage volume of 9,800 and 13,000 bbls, respectively. The seventeen stages were pumped over a period of nine days.

Table 2 Details of field test wells

		Well 1:	Well 2:	Units:
Treatment	Clean Volume	415,000	550,000	gal
	Slurry Rate	90	90	bpm
	Proppant Concentration	0-4	0-4	ppa
	Proppant Volume	450,000	660,000	lbs
	Number of stages	8	9	stages
	Fluid type	Slickwater	Slickwater	
	Casing OD, weight	5.5”, 20#	5.5”, 20#	in, lb/ft
	Maximum Treating Pressure	9,500	9,500	psi
Formation	Name	Marcellus	Marcellus	
	Type	Shale	Shale	
	Temperature	132	132	°F
	Permeability	0.01	0.01	mD
	Porosity	10	10	%

Mix water for the treatment was a combination of fresh water from local streams (80%) and flowback water (20%). The treatments were performed in February 2010, when the ambient temperature ranged from 20 to 30 °F. The covered water tanks were heated to approximmately 40 °F. As other recently fractured wells were placed on production, the flowback/PW was transported to storage tanks. Typically, PW from different nearby wells is combined in tanks and saved for the next fracturing treatment. In preparation at the new location, tanks are set specifically to hold the produced water. In this case, there were thirteen frac tanks, positioned apart from the fresh water tanks. A schematic of the wellsite is shown in Figure 4.

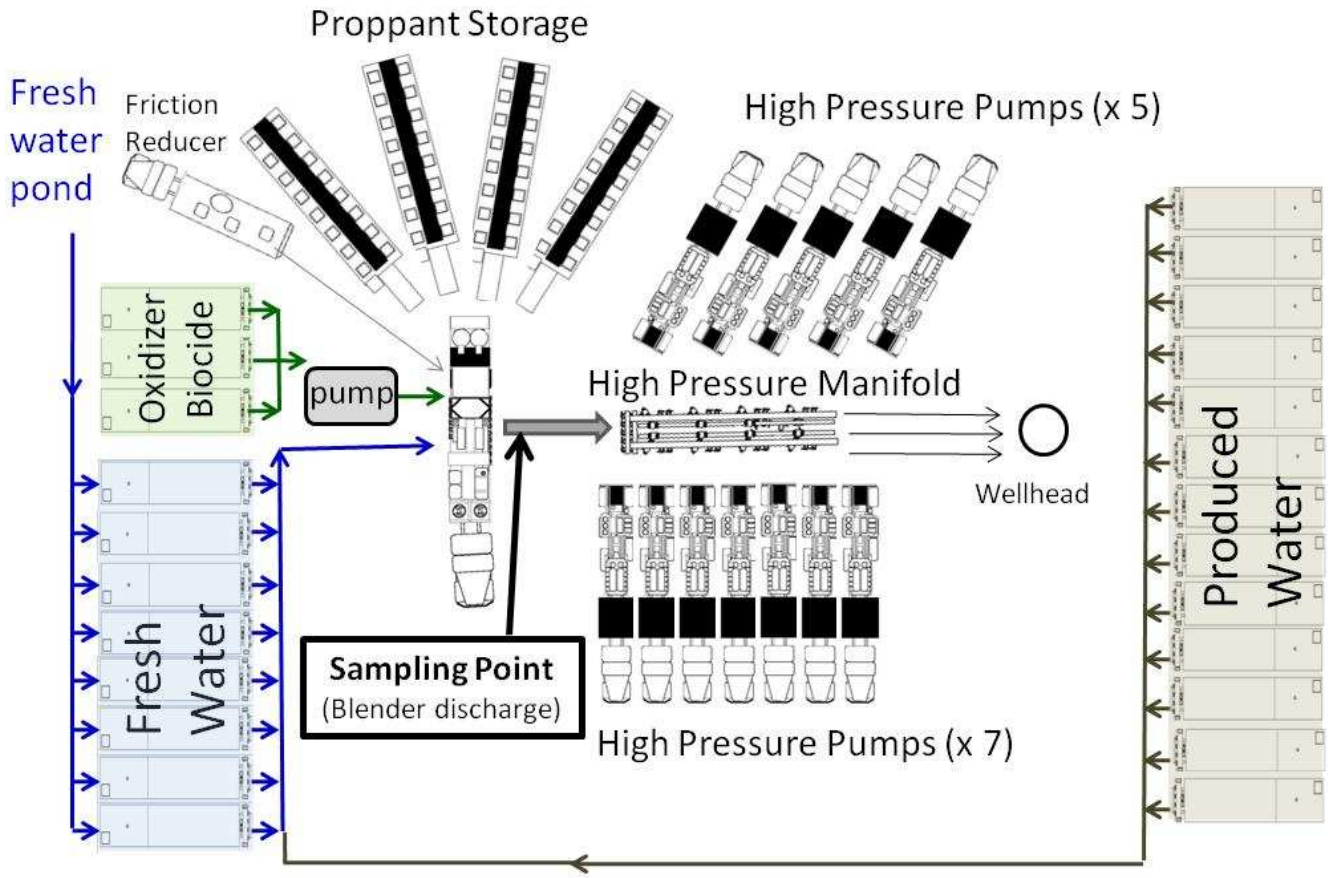

Figure 4 Schematic of the wellsite in this case study. Water was sampled from the fresh and produced water tanks, and from the blender.

The PW for this study had a pH of 5.8 and a salt concentration of 87,100 ppm TDS and was stored in 13 frac tanks spotted on location. The fresh water was obtained from a surface source. This water was stored in off site retention ponds and not treated prior to it being pumped to the 8 'working tanks' on location. The fresh water was continuously pumped into the working tanks during the stimulation treatment. The fresh water had a pH of 8.0, and a salt concentration of 3,400 ppm TDS. To limit the TDS of the water, PW was mixed with fresh water at a ratio of 1:5 (i.e. 20%). The comingled waters had 32,100 ppm TDS and 208 mg/L TOC, pH 5.73. When higher ratios of PW were attempted, the surface treating pressures significantly increased at a given rate and friciton reducer concentration.

Biocidal efficacy of the oxidative biocide against GHB, SRB, and APB was evaluated according to NACE Test Method TMO194-94. Appropriate media and containers were brought to location, and representative biological samples were obtained of the fresh water and PW on location prior to mixing to establish a baseline sample so that the starting bacterial count, reported in cells/ml, could be used for contrast against the count in the blended slickwater fluid containing the biocide. During the treatment, samples were collected from the fresh water tanks, PW tanks, mix tanks containing 80:20 mix water before the biocide was added (at the suction side of the blender), and at the discharge side of the blender after the biocide was added (side opposite the injection point). Each sample was then analyzed. After the fracturing treatment was completed, the microbiologist collected flowback water samples at 21 days, 51 days, and 81 days after the well was placed on production. The bacteria load in the PW samples was also determined. The populations of the three relevant classes of bacteria in different waters are shown in Figure 5

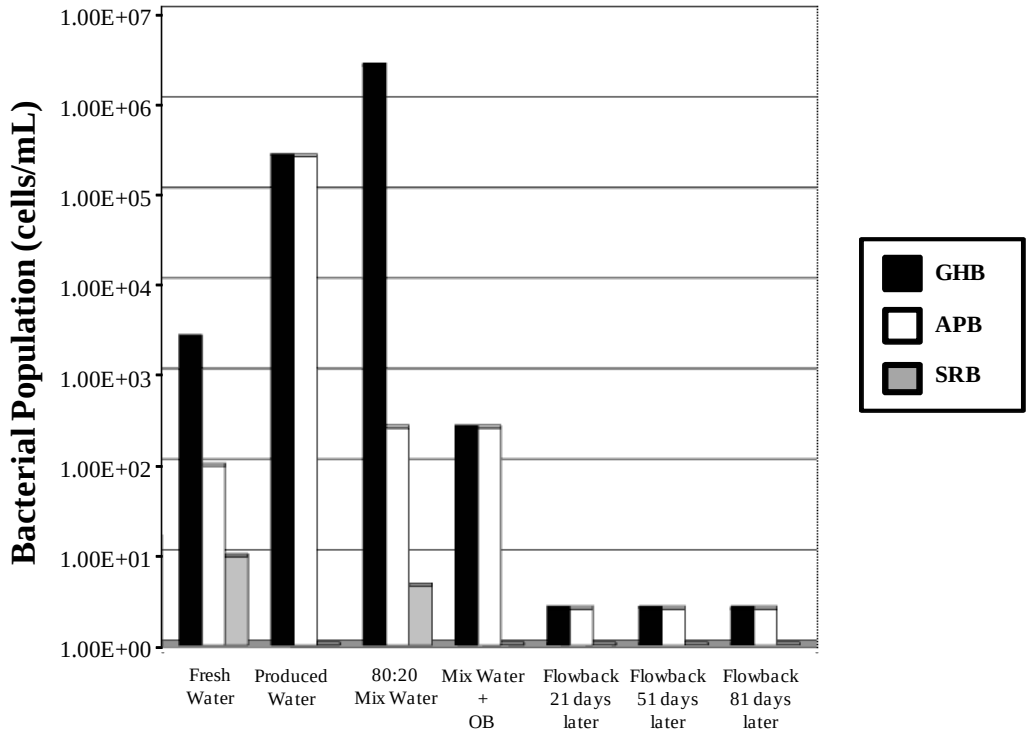

Figure 5 Bacterial test results, including fresh and produced water, an 80/20 mixture of PW and FW, and flowback samples from the well.

The bacterial counts are, in general, highest for GHB, followed by APB and SRB. Produced water contains the most bacteria, as expected. The addition of biocide decreases the number of bacteria by several orders of magnitude, and the populations are successfully controlled out to 81 days post-frac. This data demonstrates that the oxidative biocide effectively limits growth of problem bacteria when it is applied as a chemical additive in the recommended manner.

A related DGGE test was also performed on a reduced sample set to establish whether bacteria in the 21 day flowback sample were the same as bacteria in the 80:20 mix water. The general principle of this type of gel electrophoresis test is that the actual biochemical constituents of the bacteria (e.g. genetic material, proteins, cell wall material, etc.) act as a 'fingerprint' when they are forced through a gel block by an electrophoretic current. It is clear from Figure 6 that the bacteria in the 1st flowback sample at 21 days are not the same as bacteria in the frac fluid. The DGGE gel also shows that no genetic material was detected in the flowback water sample after 21 days post treatment.

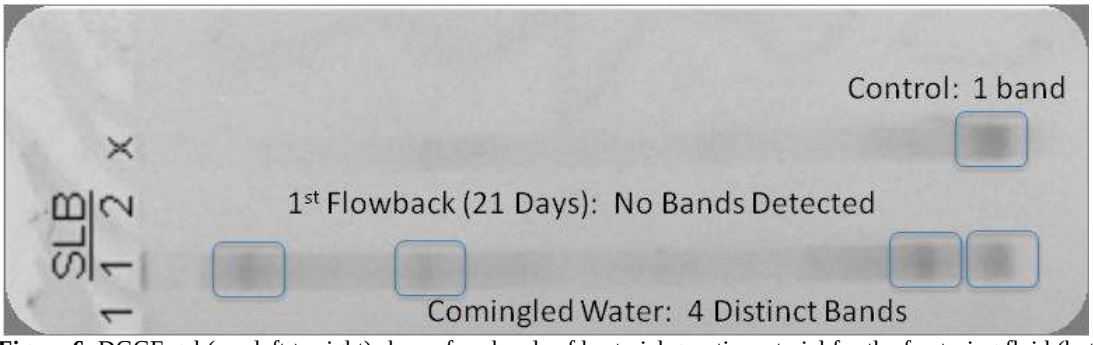

Figure 6 DGGE gel (run left to right) shows four bands of bacterial genetic material for the fracturing fluid (bottom lane, numbered "1"), but no bands for flowback water sample taken at 21 days (middle lane, numbered "2").

Conclusions

1. A fracturing fluid composed of 20% produced water that is treated with an oxidative biocide at a level sufficient to reduce bacteria to <100 cells/ml shows no bacterial re-growth in the flowback water for up to 81 days.
2. In laboratory testing, the oxidative biocide showed faster action than glutaraldehyde, a non-oxidizing biocide. In the field test, the treatment was shown to have adequate persistence.
3. The laboratory evaluation of this oxidative biocide demonstrates negligible corrosion on typical metals used in oilfield surface equipment and tubular goods.

4. This oxidative biocide is compatible with both slickwater and guar-based fracturing fluid formulations. As for any new additive, it is recommended to check compatibility of this oxidative biocide with additives and mix water prior to the stimulation treatment.
5. The oxidative biocide is added to the fracturing fluid in a form which is not a wellsite hazard in terms of health, biodegradability, and toxicity.
6. The successful use of an oxidative biocide in the field is demonstrated in this case study. Though the results are encouraging, it is suggested that additional field studies be performed to validate the robustness of this approach.

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