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A Systematic Study of Shear-Tolerant Micro-Emulsion Fracturing Fluid

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Abstract

Fluid loss control, formation damage prevention and mitigation are critical issues that affect hydraulic fracturing productivity in the low permeability gas reservoirs. In this work, we propose that micro-emulsion fracturing fluids should be used to reduce fluid loss and avoid formation damage. We used the Winsor IV single phase micro-emulsions, emulsified by non-ionic surfactants and co-surfactants, as emulsion component. Then, we mixed the emulsion with Guar Gum component, formulated a micro-emulsion fracturing fluid. Different parameters of formulated system, including gelation times, shearing tolerance at high temperature, fluid loss, formation damage and pipe friction were evaluated to characterize this novel fracturing fluid. With comprehensive measurements and evaluations, along with comparison to the conventional fracturing fluids, we have demonstrated that the micro-emulsion fracturing fluid has less fluid loss, controllable formation damage, and it can still maintain a high viscosity under high temperature for a long period. The proposed micro-emulsion fracturing fluids have the potential to solve fluid loss and formation damage problems which are affecting fracturing treatment for the high temperature and low permeability formations.

Keywords: Hydraulic fracturing, Micro-emulsions, Fluid loss, Formation damage, Shear-tolerant

Introduction

Undoubtedly, hydraulic fracturing technique and horizontal drilling techniques have changed the landscape of the world's energy production. Successful applications of hydraulic fracturing technique on the unconventional shale gas formations leveraged world energy production, especially for North America area, meanwhile creating thousands of jobs. However, innovations for fracturing operations are still demanded to reduce cost, increase well life span and secure energy supply for centuries.

Fluid loss and formation damage are two factors limiting fracturing operations since it was developed [1]. The primary concern for a fracturing treatment is fluid loss. Fluid loss issues increase the total water consumption for fracturing treatment, which puts

hydraulic fracturing under controversy and also increases cost of fracturing operation. The second problem, formation damage, reduces formation permeability, which limits well lifetime and undermines productivity.

Several new technologies have been proposed to solve these problems, such as critical state CO₂ or liquefied petroleum gas (LPG) [2] [3]. One drawback of such techniques is that they are utterly different from the conventional fracturing process, thus costing more to modify the current equipments. Visco-Elastic Surfactants (VES) fracture [4] [5] is another new technology developed to solve fluid loss problem. However, under high temperature and high shear rate conditions, fluid viscosity would be compromised, besides the high cost of VES.

Micro-emulsions, which have already been widely applied in upstream, are mixtures of oil and water with surfactants [6] [7] [8]. Applications of micro-emulsions/gel as fracturing fluids were reported by other groups [9] [10] [11]. By altering wettability of pore throat, micro-emulsion can hydrophobize rock surface and reduce capillary pressure. A lower capillary pressure is preferred in many ways, from increasing productivity to reducing trapped fluid phase. In addition, benefited from the fact that micro-emulsion fracturing fluids are still homogeneous liquid phase under ambient temperature and pressure, instruments for conventional fluids can also implement micro-emulsion fracturing fluids with minor changes, which avoids cost for new treatment systems. Last but not least, micro-emulsion has a higher surface volume ratio compared to macro-emulsion system. By virtue of large surface volume ratio, emulsion droplets have higher chance to contact with rock surface hence alter the wettability. Also diesel usage in the fluids is reduced by the large surface volume ratio, which renders the diesel fracturing fluid as an economy method for fracturing.

Here we present a systematic investigation on the application of micro-emulsion based fracturing fluids to reduce fluid loss and formation damage, aiming to provide other researchers a standard protocol to test different micro-emulsion fracturing fluid systems. To obtain a stable emulsion, we screened different compositions of water, oil and surfactants. With the aid of a ternary phase diagram (Figure 1), we located the stable Winsor IV phase micro-emulsions. Based on that, we developed micro-emulsion fracturing fluids. Fluid rheology is the most crucial parameters for fracturing fluids [12]. In our work, fluid rheological properties were measured and formation damage was evaluated with synthetic core samples. Experiment results showed that the new micro-fracturing fluids can maintain a high viscosity under high shearing rate and high temperature. Further investigations on micro-emulsion fracturing system also showed a reduced formation damage and fluid loss.

Materials and Methods

Triton-X 100 (4-(1,1,3,3-Tetramethylbutyl)phenyl-polyethylene glycol, Surfactant), Ammonium persulfate (Breaker) and n-butanol (Co-surfactant) are purchased from Sigma-Aldrich. Hydroxypropyl Guar Gum (HPGG,Viscosifier), organoboron crosslinker HYJ-2 are purchased from Shuntong Chemical, China. Encapsulated breaker OB-1 is obtained from Southwest Petroleum University, China. Diesel component is directly purchased from market (Shell, United States).

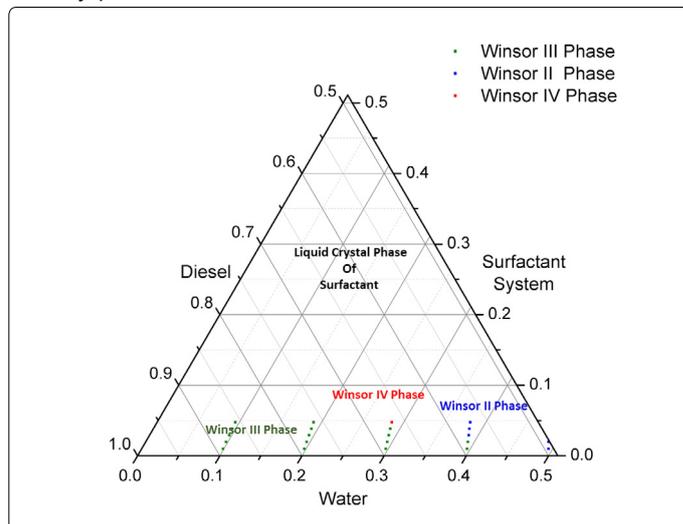


Figure 1. A tertiary phase diagram of Triton-X 100/n-Butanol, Diesel, Water mixture (zoomed into the Diesel corner). Compositions are labeled in weight ratio. Different color indicates the different Winsor phase type, see legend. Winsor II, III, IV phases were observed. The formulation of micro-emulsion fracturing fluids is based on the composition of Winsor IV emulsions.

We used diesel as a model oil phase to explore the phase behaviors of the micro-emulsion fracturing fluids. Blended surfactant serving as an emulsifier was obtained by mixing primary surfactant Triton-X 100 with co-surfactant n-Butanol under magnetic stirring with 2:1 mass ratio. The mixture was then stocked for later use. A thorough phase behavior study was carried out to locate an optimal composition for the micro-emulsion fracturing fluids. Results are shown in Figure 1 and Figure 2. Phase diagram (Figure 1) is drafted according the emulsion phase behaviors under different compositions. Formula for the single phase micro-emulsion (Winsor IV in Figure 2e and red symbol in Figure 1) can be obtained by mixing emulsifier, diesel and water in a 0.5:69.65: 29.85 weight ratio. The micro-emulsions are thermodynamically equilibrate phase and very stable. A batch of 6-months old samples were compared with fresh samples shown in Figure 2 and barely changed. Micro-emulsion forms spontaneously and requires minimal external energy input. A uniform single phase micro-emulsion can be simply acquired by shaking the sample vials manually. Emulsion size and droplet morphology were also checked by microscope (Figure 3. Insert)

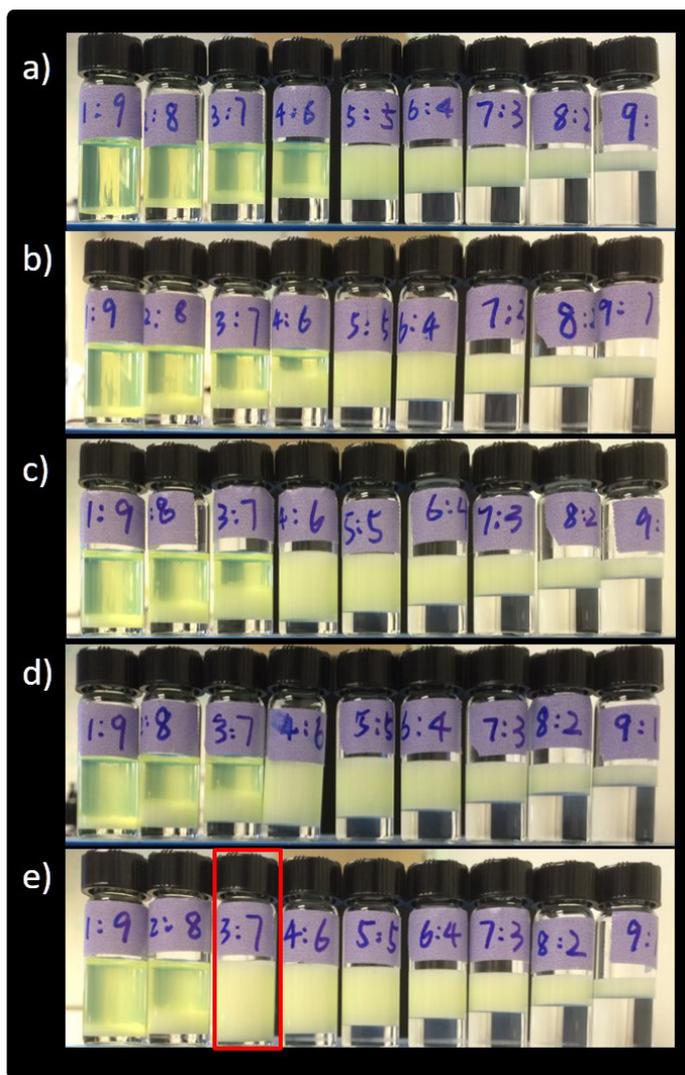


Figure 2. Phase behaviour scanning. From left to right, the oil to water ratio descends as labeled on the vials. Surfactant concentrations were 0.1%, 0.2%, 0.3%, 0.4%, 0.5% for row a), b), c), d), e), respectively. Pictures were taken after samples had been set still for 1 week. A Winsor IV type emulsion (marked as red in picture) was chosen to make the fracturing fluids.

A gel based fracturing fluid was formulated according to the formula mentioned as following. The gel based fracturing fluid consists of 2000-6000mg/L Hydroxypropyl Guar Gum (HPGG), 2000-10000 mg/L organo boron crosslinker, 1000-10000 mg/L breaker (ammonium persulfate) and 500-1000 mg/L high temperature stabilizer (alkyl alkanolamine). Within these compositions, the fracturing fluid can quickly form a gel at 40 to 100°C by vigorous mixing. Gel quality was checked visually before next step. Then the single phase micro emulsions were mixed with the gel based fracturing fluid in a 1:9 weight ratio and used for all later tests.

Interfacial tension (IFT) between the diesel oil phase and aqueous phase under influence of blended surfactants was measured with spinning drop method (M6500 Spinning Drop Tensiometer, Grace Instrument®). The IFT profile shows in Figure 3 that Triton-X/n-Butanol reaches its critical micelle concentration at 0.2 w/w% surfactant concentration, which was consistent with emulsion phase study results shown in Figure 1 and Figure 2.

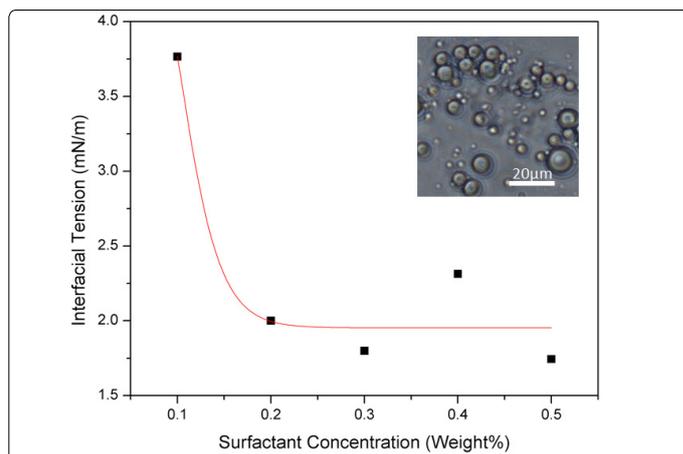


Figure 3. Interfacial tension between the diesel oil and water interface with different concentrations of surfactants. The red line represents the fitted result with lognormal model. Four data points from right side, 0.2%, 0.3%, 0.4%, 0.5%, respectively, showed interfacial tension reached a plateau. Thus we conclude the concentration of surfactant already reached their critical micelle concentration (CMC). Insert is microscopic image of micro-emulsion with 3:7 oil to water ratio and 0.5 w.t. % surfactant.

Result and Discussion

Rheological Characterization

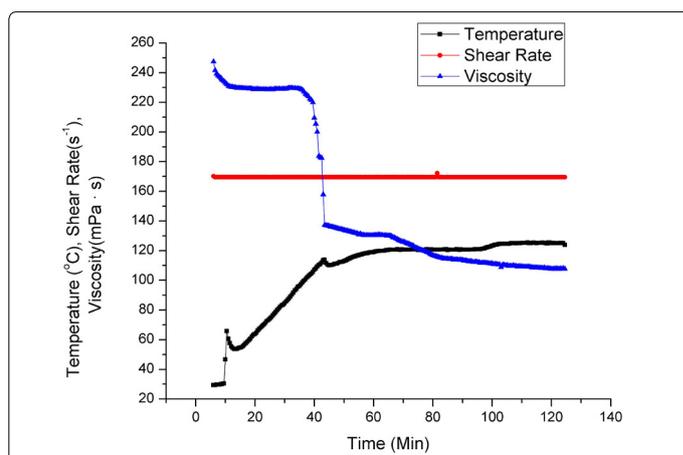


Figure 4. Rheological change of the micro-emulsion fracturing fluid under 120°C and 170s⁻¹ shearing rate.

Rheological property is tested with rheometer (HAKE Rheo Stress 600). To represent the real shearing force induced by underground formation, the shear rate is set at 170s⁻¹. Meanwhile, we introduced a gradual temperature increase to simulate the temperature increase caused by tube friction. As shown in Figure 4, fracturing fluid was sheared under a 170s⁻¹ shearing rate and temperature was gradually increased to 120°C at 5°C/min increasing rate. Due to initial temperature ramping, viscosity decreased, but after temperature reached plateau, fluid system can maintain a viscosity above 100mPa·S for more than 2 hours. This result demonstrate that our micro-emulsion fracturing fluid exhibits an excellent shear-tolerant property under high temperature.

Fluid Loss Measurement

To evaluate the fluid loss, we compared the micro-emulsion fracturing and conventional fracturing system and

measured leakoffs of different fracturing fluid systems with different micro-emulsion percentages. A lab-built leakoff measuring system is used and experiments are conducted under following conditions. Synthetic 500 PSI (3.5 MPa) differential pressure is applied on the two sides of filtration cell. Then, volume of fluid that flows out is measured with cylinder. Temperature was controlled at 90°C during the tests. Applied pressure lasted 36 minutes during the test. Experiment results were plotted in Figure 5. The synthetic core samples used in these tests were manufactured according to the composition of Sulige Eastern formation.

Result showed that a concentration of 5% or above of micro-emulsion can significantly reduce fluid loss. Although the change between 5% and 10% was subtle, we decide to use 10% concentration of micro-emulsions in our research for better demonstrations.

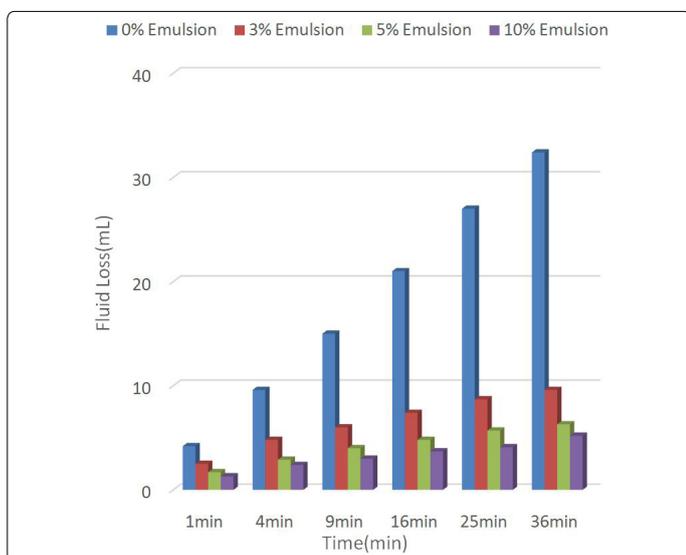


Figure 5. Fluid loss for different fracturing fluid fomulas with different micro-emulsion percentages.

Formation Damage Test

The core permeability can be calculated with Darcy-Weisbach equation,

$$K = \frac{Q \cdot \mu \cdot L}{\Delta P \cdot A}$$

Where K is sample permeability, μm^2 , Q is flow rate, ml/s, L is core sample length, cm, A is core sectional area, cm^2 , ΔP is pressure difference, MPa, and M is fluid viscosity, mPa.s.

Formation damage can be characterize by the percentage of permeability reduction:

$$\eta = \frac{K_1 - K_2}{K_1} \times 100\%$$

Where K_1 is the permeability before fracturing fluid flooding, and K_2 is the permeability after fracturing fluid flooding.

Experiments were conducted under 120°C with core flooding setup, as shown in Table 1. Data showed that the conventional guar gel fracturing fluid decreases formation permeability by 57.9%, however, our micro-emulsion fracturing fluid only decreases permeability by 16.4%. Hence we conclude

that micro-emulsion fracturing fluids only causes marginal changes in permeability compared to the conventional guar gel fracturing fluid.

Table 1. Formation Damage Test Data

Type	Core Sample No.	Length (cm)	Diameter (cm)	Porosity (%)	Permeability before test (μm^2)	Permeability after test (μm^2)	Formation damage%
Guar GelFrac	1#	4.91	2.54	14.59	1.469×10^{-3}	0.619×10^{-3}	57.9
Micro-Emulsion Frac	2#	5.73	2.54	14.6	2.647×10^{-3}	2.212×10^{-3}	16.4

Tube Friction Test

Tube friction test was carried out by a coiled tubing system with 0.001kPa pressure resolution. The total length of the coil was 3 meters. We used two different inner diameter tubes, 0.46 cm and 1.01 cm, respectively, and each test last 2 hours. The micro-emulsion fracturing fluids formulated according to previous paragraph were then injected. Also we tested the fluids under two different temperatures, 40°C and 80°C, respectively. Results showed in Figure 6. The purpose of these experiments is to provide friction reference data for later field applications.

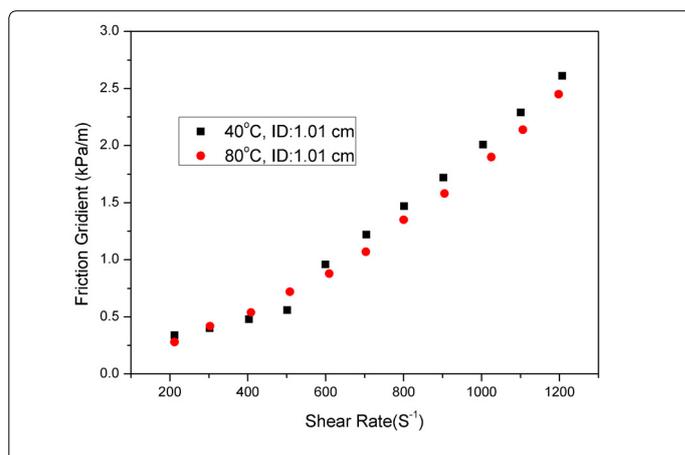
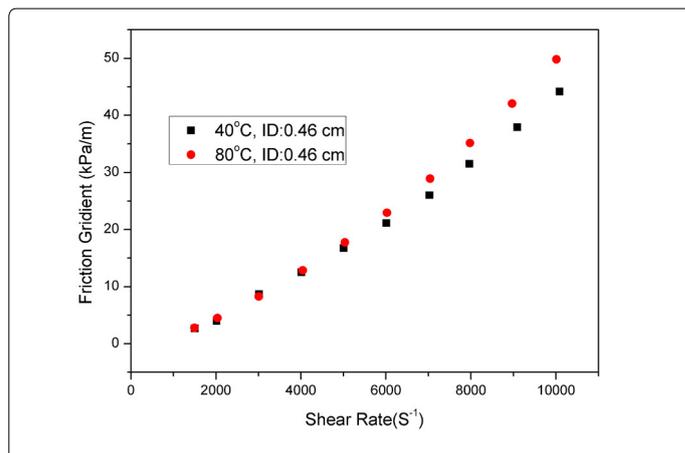


Figure 6. Tube friction profiles with 0.46cm, 1.01cm tubings, under 40°C and 80°C.

Compatible Breaker Systems

The gel breaking time can be controlled by adding different amount of breakers. Depending on the amount of breaker, it usually takes 60 to 120 minutes for the breaking

process to complete. Two different breaking chemicals are tested, namely, ammonium persulfate and encapsulated breaker. Both chemicals are compatible with our fracturing system and the breaking times for different chemicals are similar.

Conclusion

To solve the fluid loss and formation damage problems during fracturing operations, we developed a micro-emulsion based fracturing fluids. The ternary phase diagram was used to locate the optimal composition of the fracturing fluids. After optimizing fluid composition, rheological characterization was measured to access the fluid shear tolerance. Fluid loss and formation damage tests were also carried out to evaluate the fracturing fluids. Results showed that our micro-emulsion fracturing fluids have a robust shear resistance, which ensures a high operation fluid viscosity after pumping down fluid into formation. In addition, our micro-emulsion fracturing fluids caused less fluid loss and formation damage comparing to conventional guar gum fracturing fluids. Compatible breaker system was suggested here for consideration in field implementation. Tube friction profile was measured to provide a reference for field applications. The intrinsic properties of micro-emulsions and micro-emulsion fracturing fluids make them promising tools for economy fracturing operation with increased production lifetime. Also, this comprehensive research work on micro-emulsion fracturing fluid explored a standardized protocols for the later research.

Nomenclature

Visco-Elastic surfactant (VES)
Liquefied petroleum gas (LPG)
Hydroxypropyl Guar Gum (HPGG)
Interfacial tension (IFT)

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