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Characterizing the Hydraulic Fracturing Fluid Modified with Nano Silica Proppant

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Abstract

The composition of the fracturing fluid is important to not only fracture the rocks but also provide for efficient transport and placement of proppant into fractured rocks to keep it opened to extract the oil and gas. In this study, the effect of 1% nano silica proppant on the rheological properties, fluid loss and electrical resistivity of the fracturing fluids and transport characteristics in the pre-cracked sandstone were investigated at various temperatures and pressures up to 85°C and 700 psi (splitting tensile strength of rock) respectively. Two different mixes of the fracturing fluids were developed and used in this study to investigate the effects of nano silica proppant. The amount of water in the fracturing fluid varied from 90% to 93% (by the total weight of the fracturing fluid) and the percentage of the fine sand used varied from 5% to 9% (by the weight of fluid) with 1% of guar gum. Additional of 1% of nano silica increased the electrical resistivity by 18% at room temperature. With increasing the temperature from 25°C to 85°C, the electrical resistivity of fracturing fluid with and without nano silica decreased from 4.56 to 3.11 Ω -m and from 3.75 to 2.78 Ω -m respectively.

The nano silica modification increased the yield stress (τ_0) by 12% at room temperature. The yield stress of the fracturing fluid decreased by 32%, when the temperature was increased from 25°C to 85°C. The viscosity of the fracturing fluid increased by 10% with the addition of 1% of the nano silica at room temperature. A new test protocol was developed using pre-cracked sandstone to evaluate the performance of the fracturing fluids. In the range of 690 to 750 psi the discharge of the fluid increased by 30% when the fracturing fluid was modified with nano silica and the temperature was increased from 25°C to 85°C at 700 psi. The apparent permeability of the rock increased by 19% when the fracturing fluid was modified with 1% nano silica at temperature of 85°C and pressure of 700 psi. The fluid loss and shear thinning behavior of fracturing fluid with and without nano silica was quantified using a new hyperbolic model.

The results showed that the hyperbolic model predicated the fluid loss with the time, temperature, pressure and the shear thinning relationship between the shear stress with shear strain rate of fracturing fluids very well. The results also showed strong influence of the nano silica and temperature on fracturing fluid rheology, fluid loss and the fracturing behavior of the rock.

Introduction

Hydraulic fracturing which started in US in late 1940s is a technique used in various applications including the petroleum industry to free oil and natural gas trapped underground in low permeability rock formations by injecting a fluid under high pressure in order to crack the formations. The composition of a fracturing fluid varies with the nature of the formation, but typically contains 99% of water and proppant sand to keep the fractures open and a small percentage of chemical additives (Murrill and Vann 2012). The quality of fracturing fluid can be effectively maintained by continuously measuring fluid characteristics in the field and controlling its viscous properties by modifying fluid additives and injection rate. Minimizing formation damage and fracture damage is regarded as a unique goal of hydraulic fracture design (Bouts et al. 1997).

In the United States shale gas and oil production has grown rapidly in the past years with continuous technological developments in hydraulic fracturing. Hydraulically fracturing rocks increases the permeability by opening, connecting and keeping open pre-existing or new fractures in the formation. The design of the fracturing fluid is therefore critical for the success of the operation. Its main function is to open the fractures and to transport and keep the proppant along the length of the cracked rocks. The rheological properties of the fluid are usually considered the most important parameters (Economides and Nolte 1989). However, the fracturing fluid must exhibit other important properties such as minimizing fluid loss and demonstrate low friction pressure during pumping (API RP39 1998).

Also enhancing the sensing properties will help in monitoring the changes and contamination in various materials including fracturing fluids (Vipulanandan et al. 2004 - 2014).

Rock Characterization

Various types of rocks (sandstone, mudstone, coal, kota stone and shale) are encountered during hydraulic fracturing of rocks. Hence there is interest in investigating the correlation between the rock properties.

(a) Strength

Based on the literature data, the correlation between compressive strength (σc) and tensile strength (σt) was developed (Eqn. 1). Total of 73 data of σc and σt for different type of rocks were collected from various research studies as shown in Fig. 2.

The relationship for the data collected can be represented as follows:

$$\sigma_t = \frac{\sigma_c}{8.21 + 0.0003 * \sigma_c}$$
(1)

The σc of collected data varied from 1190 to 25000 psi. The σt of rock samples varied from 145 to 2052 psi. The σc and σt of the sample used in this study 9000 psi and 690 psi respectively. The coefficient of determination (R²) of Eqn. 1 was 0.82.

(a) Permeability (k)

Based on the data collected from the literature, the correlation between compressive strength (σ_c) and permeability (k) was developed (Eqn. 2). Total of 21 data of σ_c and κ for different type of rocks were collected from various research studies as shown in Fig. 3. The k values varied from $2.2*10^{-11}$ to $9*10^{-9}$ mD. Based on the experimental and literature data, the correlation between compressive strength (σ_c) and the permeability (k) was developed as shown in Fig. 3. The relationship for the data collected can be represented as follows:

$$\sigma_c = (7100 + \frac{10^{12} * k}{0.06 + 10^7 * k})$$
.....2(a)

In this study, a constant head permeability test was performed on 2.3"diameter and 3.3" long sandstone rock samples according to the CIGMAT Permeability Testing Standard using the double ring permeameter (Fig. 12). The water was allowed to flow through the rock under a pressure of 100 psi to measure the permeability. The coefficient of determination (R^2) for Eqn. 2(a) was 0.92. The permeability (k) can be represented in terms of compressive strength (σ_c) as follows:

$$k = (\frac{(\sigma_c - 7100) * 0.06}{10^{12} - (\sigma_c - 7100) * 10^7}) \dots 2(b)$$

Types of Hydraulic Fracturing Fluids

Hydraulic fracturing treatment involves pumping a proppant free viscous fluid which is usually water with some fluid additives, in order to generate high viscosity so that it can be used for fracturing the rock faster with limited amount of fluid that can escape into the formation. This causes the pressure to rise and the rock to break creating fractures or

enlarging existing ones. After fracturing the formation a propping agent such as sand is added to the fluid. The slurry that is pumped into the newly formed fractures in the formation must prevent them from closing when the pumping pressure is released. The proppant transportability of a base fluid depends on the type of viscosity modifying additives that have been added to the water (Lukocs et al. 2007).

The mechanics of hydraulic fracturing is a convenient description of the processes and mechanisms that are important to fracturing technology. Mechanics generally refers to an engineering discipline that is concerned with the mechanical properties of the material under consideration and the response of that material to the physical forces of its environment. Hydraulic fracturing is complicated because it involves four different types of mechanics: fluid, solid, fracture and thermal. In fracturing, fluid mechanics describes the flow of one, two or three phases within the fracture; solid mechanics describes the deformation or opening of the rock because of the fluid pressure; fracture mechanics describes all aspects of the failure and parting that occur near the tip of the hydraulic fracture; and thermal mechanics describes the exchange of heat between the fracturing fluid and the formation.

To develop tools for the design and analysis of a process as complicated as hydraulic fracturing, it is necessary to build models that describe each of the responses sufficiently. The fracturing fluid contains suspended proppant particles that are to be placed in the fractures to prevent them from fully closing once the hydraulic pressure is released. This process forms conductive channels within the formation through which hydrocarbons can flow. Once at least one fracture is created and at least a portion of the proppant is substantially in place, the viscosity of the fracturing fluid may be reduced to remove it from the formation. In certain circumstances a portion of the fracturing fluid may be lost, through undesirable leak off into natural fractures present in the formation. This is problematic because such natural fractures often have higher stresses than those created by a fracturing operation. These higher stresses may damage the proppant and cause it to form an impermeable plug in the natural fractures which may prevent hydrocarbons from flowing through the natural fractures (Welton et al. 2010).

Guar gum polymer it is one of the polysaccharides that has been used to increase the viscosity of fracturing fluids. However the conductivity of many fractures created with guar-based polymers is low because of residual unbroken polymer gel remaining in the fracture (Xu et al. 2011). Since the late 1950s, more than half of fracturing treatments have been conducted with fluids comprising of guar gums, or guar derivatives such as hydropropyl guar (HPG), carboxymethyl guar, and carboxymethyl hydropropyl guar. Xanthan gum and scleroglucan gum have also been shown to have excellent proppant suspension ability, but they are more expensive than guar derivatives and therefore used less frequently. Polyacrylamide (PAM) and polyacrylate polymers and copolymers are typically used for high temperature applications or as friction reducers at low concentrations for

all temperatures range (Lukocs et al. 2007).

Nanomaterials

Nanomaterials are excellent tools for the development of sensors and imaging contrast agents due to the significant alterations in their optical, magnetic and electrical properties (in comparison to their bulk analogues) along with their ability form (electrically and/or geometrically) percolated structures at low volume fractions Krishnamoorti (2006). Such nanomaterials, when combined with smart fluids, can be used as extremely sensitive down hole sensors for temperature. pressure and stress even under extreme conditions. Nanoparticles have been successfully used in drilling fluids for the past 50 years. Only recently all the other key areas of the oil industry such as exploration, primary and assisted production, monitoring, refining and distribution are approaching nanotechnologies as the potential philosopher's stone for facing critical issues related to remote locations (such as ultra-deep water and artic environments), harsh conditions (high-temperature and high-pressure formations), nonconventional reservoirs (heavy oils, tight gas, tar sands) (Matteo et al. 2012).

Nano Silica has been used or considered for use in many applications and it has received increasing attention also in building materials, with potential advantages and drawbacks being evaluated (Berra et al. 2012). The addition of nano silica to cementitious mixes produces a remarkable reduction of the mix workability, due to instantaneous interactions between the nanosilica and the liquid phase of the cementitious mixes, with formation of gels characterized by high water retention capacities. The delayed addition of mixing water proves to be an effective way of reducing the adverse effect of nano silica on mix workability, without changing the water/binder ratio and/or adding super plasticizer. In contrast, no workability (ii) improvement associated with delayed water addition was observed for Portland cement mixes (Berra et al. 2012).

The development of nano silica based high performance concrete will possibly help reducing the cement consumption for specific grade of concrete. The reduction in cement usage will help in protecting the environment to a great extent (Berra et al. 2012).

Fluid Loss Additives

Fluid loss control is essential for an efficient fracturing treatment. Several types of materials were used to provide fluid loss control, but the effectiveness of the various types depends on the type of fluid loss problem such as loss to low or high permeability matrix or loss to micro-fractures. During leak off into the rock matrix, fluid enters the pore spaces of the rock. Some polymers such as guar gum are filtered out on the surface of low permeability rocks. A fluid containing that polymer is called wall building fluids because of the layer of polymer and particulates that builds up on the rock. This layer called a filter cake is generally much less permeable than the formation. If the fluid contains particulates of the proper size, these particulates tend to plug the pore spaces and enhance the formation of filter cake.

In high permeability formations, polymer and additives may be able to penetrate most pore throats and form an internal filter cake. In this case, most of the resistance to leak off, and therefore pressure drop, occurs inside the rock, leaving only a small fraction of the total pressure drop in the external cake. The yield stress of a polymer cake depends on the polymer concentration and pressure gradient in the cake, whereas the shear stress of the fluid is determined by the rheological properties of the fluid and the shear strain rate (\dot{r}) at the formation face. Silica flour has been shown to be an effective fluid loss additive for helping establish a filter cake (Navarrete and Mitchell 1995).

Objectives

The overall objective was to investigate the effect of nano silica proppant on the performance of the hydraulic fracturing fluid. The specific objectives are as follows:

- (i) Evaluate the effect of nano silica on the rheological, electrical resistivity (nondestructive and sensing properties) and fluid loss properties of the fracturing fluid at different temperatures.
- (ii) Evaluate the effect of nano silica on the fracturing fluid transport through pre fractured sandstone.

Materials and Methods

(i) Sand

Uniformly graded sand was used in this study. It had a coefficient of uniformity (C_u) of 2.53, the coefficient of gradation (C_c) of 0.90 and 50% of the particles were passing 0.46 mm sieve ($d_{50} = 0.46$ mm) and hence the average specific surface area was 0.002 m²/g. Specific gravity of the sand was 2.65.

(ii) Rock (Sandstone)

Field rock samples were used for hydraulic fracturing test. Permeability, water absorption, unconfined compressive strength and spilt tension tests were performed according to ASTM Standards. These results are summarized in Table 1.

(iii) Nano Silica

Nano silica with average grain size of 20 nm and specific surface area of $100 \text{ m}^2/\text{g}$ was used in this study. The specific surface area of the nano silica was over 50,000 times higher than the sand used in this study.

(iv) Guar Gum

Guar gum (HPG) with a specific surface area of 22 m²/g and the density of 0.55 gm/cm³ was used.

Methods

(i) Rheological Properties

The rheological properties such as shear stress - shear strain rate and viscosity (μ) for fracturing fluids were measured using a viscometer. Two different mixes with and without nano silica were used as summarized in Table 2. The fracturing fluids were tested in the temperature ranged from 25

to 85 $^{\circ}$ C using a viscometer with the speed range of 0.3 to 600 rpm.

(ii) Electrical Resistivity of Fracturing Fluid

Two different resistivity devices were used to measure the electrical resistivity of fracturing fluid. API resistivity meter measured the resistivity of fluids, slurries and semi-solids with resistivities in the range of 0.01 to 400 Ω -m. Conductivity meter was used to compare the results with resistivity in the range of 0 to 199.9 $\mu \text{S/cm}.$ Both of the devices were calibrated using standard solution of sodium chloride (NaCl). The electrical resistivity of the fracturing fluid with and without nano silica was measured for each 10 $^{\circ}\text{C}$ temperature interval.

(iii) HTHP Filtrate Measurement

Measuring the HTHP fluid loss of a fracture fluid involves heating the fluid in a controlled environment to a temperature that is expected in the well. When test temperature was reached, long term filtrate volume and cake thickness was determined at a temperature differential to simulate downhole conditions. The equipment designed for this purpose includes a heating jacket (with a bimetallic thermostat) a cell to contain the fluid, a means to pressurize the cell and a means of collecting filtrate.

Gauging the effect of temperature on the fracturing fluid filtrate volume is the main purpose of the HTHP test and accurate temperature measurements are required. Thermocouple device was used to monitor the fluid temperature the fluid in the cell. Test results indicated the fluid temperature met the targeted test temperature within the API recommended one hour heat up period for the 500 mL HTHP cell. The filtrate volume was measured according to API specification 13A.

The average of thickness of filter cake at the end of the test was measured using a Vernier caliper.

Proposed Hyperbolic Model

It is important to quantify the changes in the hydraulic fracturing fluid properties due to the additives and environment (temperature, pressure). The hyperbolic model has been used for different applications under different conditions. Vipulanandan et al. (2007) used hyperbolic relationship to represent the variation of in-situ vertical stress and logarithmic undrained shear strength of the soft marine and deltaic clays. This relationship better represented the marine clay as compared to the deltaic clay. Usluogullari et al. (2012) used hyperbolic relationship to represent the compressive strength variation with curing time. Similar trend was observed between curing time and elastic modulus. Nonlinear relationships were developed to represent the changes in properties with curing time and cement content. Mohammed and Vipulanandan (2013) used the hyperbolic relationship to predicate the relation between compressive and tensile strength of sulfate contaminated CL soils with and without polymer treatment.

Based on the inspection of the test data following relationship was proposed:

$$Y = Yo + \frac{X}{A + R * X} \dots (3)$$

Where:

X: time or shear strain rate or temperature (independent variable).

Y: is the fracture fluid property with varying X value.

Y_o, A and B: model parameters.

Relationship proposed in Eqn. (3) can be used to represent various linear and nonlinear trends based on the values of the parameters A and B as shown in Fig.1.

Results and Analyses Fracturing Fluid

(a) Shear Stress -Shear Strain Rate Relationship

The shear stress –shear strain rate relationship for the fracturing fluid with 9% sand (Mix 1) is shown in Fig. 4 (a). The shear stress at shear strain rate of 1024 sec⁻¹ reduced from 126.8 Pa to 90.9 Pa when the temperature was increased from 25°C to 85°C. The shear stress – shear strain rate relationship for the fracturing fluid with 5% sand and 1% nano silica (Mix 2) is shown in Fig. 4 (b). The shear stress at shear strain rate of 1024 sec⁻¹ reduced from 183.4 Pa to 120.5 Pa when the temperature was increased from 25°C to 85°C. Additional of nano silica increased the shear stress at a strain rate of 1024 sec⁻¹ in the fracturing fluid by 44%.

The apparent viscosity at a strain rate of 170 sec⁻¹ and temperature of 25°C for the fracturing fluid with 9% sand (Mix 1) was 522 cP and it reduced to 206 cP at temperature of 85°C, a 61% reduction. With the increase in temperature, the apparent viscosity at a strain rate of 170 sec⁻¹ and temperature of 25°C for the fracturing fluid with 1% nano silica and 5% sand (Mix 2) was 574 cP and it reduced to 226 cP at temperature of 85°C, a 61% reduction. The relations between shear stress and shear strain rate were conducted on the fracturing fluids at different temperatures up to 85°C. The results were predicated using Eqn 3 as shown in Fig. 4. With the increasing the temperature the shear stress decreased. Increasing the nano silica to 1% the shear stress at shear strain rate of 1024 sec⁻¹ increased by 44% at room temperature. With increasing the temperature for the fluid with 1% nano silica to 85°C, the shear stress at shear strain rate of 1024 sec⁻¹ reduced by 58% as shown in Fig. 4(b).

The model parameters A and B with coefficient of determination (R²) are summarized in Table 3. Parameter A varied from 1.28 to 10.55 when the temperature increased from 25°C to 85°C and the parameter B varied from 0.013 to 0.025 when the temperature increased from 25°C to 85°C.

(b) Yield Stress (τ_0)

Yield stress (τ_o) was measured according to API specifications based on Bingham plastic model. Yield stress (τ_o) of fracturing fluid increased from 87.4 Pa to 98.3 Pa when the nano silica content changed from 0 to 1% at T=25°C as

shown in Fig.5. For the fracturing fluid using 9% sand (Mix 1) the τ_o reduced by 36% when the temperature increased from 25 to 85 °C and fracturing fluid using 1% nano silica and 5% sand (Mix 2) the τ_o decreased from 98.3 to 67 Pa with increasing the temperature from 25°C to 85 °C.

The relation between yield stress (τ_o) and temperature was predicated using Eqn. 3 as shown in Fig. 5 and the model parameters with coefficient of determination (R^2) are summarized in Table 4. Additional of nano silica increased the yield stress in the fracturing fluid by 12%.

(c) Viscosity (μ)

Viscosity (μ) is the slope of shear stress (τ) and shear strain rate (\rlap/r) relationship at a selected shear strain rate. Addition of 1% nano silica increased the viscosity of fracturing fluid at a shear strain rate of 170 s⁻¹ by 10% at T=25°C as shown in Fig.6. For the fracturing fluid using 9% sand (Mix 1) the μ at a shear strain rate of 170 s⁻¹ of reduced from 479 to 178 cP when the temperature increased from 25 to 85 °C. Fracturing fluid using 1% nano silica and 5% sand (Mix 2) the μ decreased from 526 to 192 cP with increasing the temperature from 25 to 85°C. The relation between viscosity (μ) at shear strain rate of 170 s⁻¹ and temperature was predicated using Eqn. 3 as shown in Fig. 6 and the model parameters with coefficient of determination (R²) are summarized in Table 4.

Additional of 1% nano silica and decreasing the sand content by 4% increased the apparent viscosity at a shear strain rate of 170 sec⁻¹ in the fracturing fluid by 11% at 25°C.

(d) Electrical Resistivity

Addition of 1% nano silica to the fracturing fluid increased the electrical resistivity. Also increasing the temperature the electrical resistivity of fluids nonlinearly decreased. Adding 1% nano silica increased the electrical resistivity of the fluid from 3.75 to 4.56 Ω -m at room temperature, an increasing in resistivity of 18% as shown in Fig. 7. For the 9% sand (Mix 1) the electrical resistivity decreased from 3.75 to 2.78 Ω -m when the temperature was increased from 25 to 85°C, but in the 1% nano silica and 5% sand (Mix 2) the electrical resistivity changed from 4.56 to 3.11 Ω -m when the temperature increased from 25°C to 85°C as shown in Fig.7. Equations (3) and (4) were developed to predict the electrical resistivity (ρ) and temperature (T) relationship of fluids with and without nano silica.

Mix 1 (9% sand):

$$\rho = 3.75*(1+8.4*(T-To)^{-0.25})....(4)$$

Mix 2 (1% nano silica with 5% sand):

$$\rho = 4.56 * (1 + 12.6 * (T - To)^{-0.3}) \dots (5)$$

Where:

To: room temperature (To=25°C).

 ρ : electrical resistivity of fracture fluid at different temperatures (85°C \geq T \geq 25° C).

The electrical resistivity is a good tool to use as quality control for the hydraulic fracturing fluid. The coefficient of determination (R²) of Eqn. 4 and Eqn. 5 were 0.98 and 0.95 respectively.

(e) Filtration Loss

Long term fluid loss test on the fracturing fluids was performed and the results are shown in Fig. 8. Based on the Eqn.1, the volume of fluid loss at 30 minutes was measured as shown in Fig. 9. With the additional of the 1% nano silica, the filter volume of the fracturing fluid decreased by 60% at room temperature. The volume of fluid loss of the fracturing fluid using 9% sand (Mix 1) increased by 52% when the temperature increased from 25 to 85°C. For the fracturing fluid using 1% nano silica and 5% sand (Mix 2), the volume of fluid loss increased by 76% when the temperature was increased from 25 to 85°C as shown in Fig. 9.

The parameters A and B for fluid loss were influenced by the sand content (S), water content (W), polymer content (P), nano silica content (NS) and temperature (T) °C. The Eqn. 6 proposed linear model was analyzed using multiple regression method (least square). The linear relationship is as follows: Model Parameters (A or B)

$$= k + a * (S) + b * (W) + c * (P) + d * (NS) + e * (T) \dots (6)$$
Where:

k, a, b, c, d and e are model parameters and were determined by multiple regression analyses of the test data are summarized in Table 5.

(f) Filter cake

The thickness and resistivity of the filter cakes for two fracturing fluids were measured at the end of tests. With the additional of the 1% nano silica, the thickness of filter cake of the fracturing fluid increased by 30% at room temperature as shown in Fig.10. The thickness of filter cake of the fracturing fluid using 9% sand (Mix 1) increased by 50% when the temperature was increased from 25°C to 85°C. The thickness of filter cake of the fracturing fluid using 1% nano silica and 5% sand (Mix 2), increased by 45% when the temperature was increased from 25°C to 85°C as shown in Fig.10. The resistivity of the filter cake reduced from 4.02 to 3.84 Ω -m with the additional of 1% of nano silica at room temperature.

The resistivity of filter cake for fracturing fluid using 9% sand (Mix 1) and 1% nano silica and 5% sand (Mix 2) reduced by 25% and 31% respectively when the temperature was increased from 25°C to 85°C as shown in Fig.11.

In this study, four different fracturing tests setup were tested. The sandstone rock sample was saturated and precracked using splitting tensile test.

Rock- Fracture Fluid Interaction

In this study, four different rock – fluid interaction tests were performed. The sandstone rock samples were saturated and pre-cracked using splitting tensile test.

(a) Room Temperature (T=25°C)

Test # 1: the fracturing fluid used in this test was 9% sand (Mix 1); the fracturing fluid was subjected to different pressures up to 700 psi at room temperature. The amount of the fluid collected from the sample was zero mL at 100 psi for 26 hours, by increasing the pressure to 500 psi and 700 psi the discharge collected increased to 33 mL and 72 mL respectively as shown in Fig. 13. The permeability of the rock increased by 89% and 103% under pressure of 500 psi and 700 psi respectively as shown in Fig.14.

Test # 2: the fracturing fluid used in this test was 1% nano silica and 5% sand (Mix 2) and the effect of adding 1% nano silica at room temperature was investigated. The amount of the fluid collected from the sample was zero mL at 100 psi after 22 hours, by increasing the pressure to 500 psi and 700 psi the discharge collected increased by 36% and 27% respectively as shown in Fig. 13. The permeability of the rock also increased by 25% and 37% when the pressure changed from 100 psi to 500 psi and 700 psi respectively as shown in Fig. 14. Adding 1% nano silica increased the permeability of the rock at room temperature by 99%, 25% and 37% under 300 psi, 500 psi and 700 psi respectively as shown in Fig. 14.

(b) Higher Temperature (T=85°C)

Test # 3: the fracturing fluid used in this test was 9% sand (Mix 1) at a temperature of 85°C. The amount of the fluid collected from the sample increased from 0 to 18 mL at 100 psi after 24 hours, by increasing the pressure to 500 psi and 700 psi the discharge collected increased by 24% and 7% respectively as shown in Fig. 13. The permeability of the rock also increased by 25% and 37% when the pressure changed from 100 psi to 500 psi and 700 psi respectively as shown in Fig.14.

Test # 4: the fracturing fluid used in this test was 1% nano silica and 5% sand (Mix 2) and the effect of the 1% nano silica at T=85°C temperature was investigated. The amount of the fluid collected from the sample increased from 0 to 7 mL at 100 psi at 18 hours, by increasing the pressure to 500 psi and 700 psi the discharge collected from the mid valve increased by 43% and 27% respectively as shown in Fig. 13. The permeability of the rock also increased by 25% and 37% when the pressure changed from 100 psi to 500 psi and 700 psi respectively as shown in Fig.14. Adding 1% of the nano silica increased the permeability of the rock at T=85°C by 15%, 14% and 14% under 300 psi, 500 psi and 700 psi respectively as shown in Fig. 14.

Conclusions

In this study, rheological properties, electrical resistivity and fluid loss of nano silica modified fracturing fluid was investigated. Also the sandstone rock – fluid interaction was investigated at varying temperatures and pressures up to the splitting tensile strength of the rock. Based on the experimental study and modeling following conclusions are advanced:

- 1. Electrical resistivity of the fracturing fluid increased with the addition of nano silica. Resistivity decreased with increasing the temperature and it can be used as a good tool for quality control of the fracturing fluid.
- 2. Yield stress (τ_o) of fracturing fluid increased with increasing of nano silica content. Increasing the nano silica content in the fracturing fluid to 1% increased the yield stress by 10% at room temperature.
- 3. Addition of 1% nano silica and reducing the sand content by 4% in the fracturing fluid decreased the fluid loss by 16% and 18% at temperatures of 25°C and 85°C respectively.
- 4. Adding 1% nano silica and reducing the sand content by 4% in the fracturing fluid increased the permeability of the rock under different pressure and temperature.
- 5. The hyperbolic model was effective in predicting the rheological properties temperature, shear stress shear strain rate and fluid loss- time relationships.

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References

- 1. Bouts, M.N., Trompert, R.A., Samuel, A.J. (1997). "Time delayed and low-impairment fluid-loss control using a succinoglycan biopolymer with an internal acid breaker". SPE J. Vol. 2, No. 4, pp. 417–426.
- Berra, M., Carassiti, F., Mangialardi, T., Paolini, A. and Sebastiani, M. (2012). "Effects of nanosilica addition on workability and compressive strength of Portland cement pastes" Construction and Building Materials, Vol. 35, pp. 666–675.
- 3. Economides, M. J., and Nolte, K. G. (1989). "Reservoir Stimulation", Second edition. Houston, TX, Schlumberger Educational Services.
- 4. Krishnamoorti, R. (2006). "Extracting the Benefits of nanotechnology for the Oil Industry." Journal of Petroleum Technology, Vol. 58, No. 11, pp. 24-26.
- Lukocs, B., Mesher, S., Wilson, T.P.J., Garza, T., Mueller, W., Zamora, F., Gatlin, L.W., (2007). "Nonvolatile phosphorus hydrocarbon gelling agent". US Patent Application 20070173413, assigned to Clearwater International, LLC.
- Matteo, C., Candido, P., Vera, R. and Francesca, V. (2012)
 "Current and Future Nanotech Applications in the Oil Industry" American Journal of Applied Sciences, Vol. 9, No. 6, pp. 784-793.
- 7. Murrill, B. and Vann, A. (2012). "Hydraulic Fracturing: Chemical Disclosure Requirements" CRS Report for Congress, pp.1-23.
- 8. Mohammed, A. and Vipulanandan, C. (2013)." Compressive and Tensile Behavior of Polymer Treated Sulfate Contaminated CL Soil" Journal of Geotechnical and Geological Engineering, Vol. 32, No.1, pp.71-83.

- Navarrete, R.C., Mitchell, J.P. (1995). "Fluid-loss control for high-permeability rocks in hydraulic fracturing under realistic shear conditions". In: Proceedings Volume, SPE Production Operations Symposium, Oklahoma City, pp. 579–591.
- Usluogullari, O. and Vipulanandan, C. (2011)." Stress-Strain Behavior and California Bearing ratio of Artificially Cemented Sand" Journal of Testing and Evaluation, Vol. 39, No. 4, pp. 1-9.
- Vipulanandan, C., Ahossin, Y.J., and Bilgin, O. (2007)." Geotechnical Properties of Marine and Deltaic Soft Clays." GSP 173 Advances in Measurement and Modeling of Soil Behavior, pp.1-13.
- Vipulanandan, C. Heidari, M., Qu, Q., Farzam, H. and Pappas, J. (2014)."Behavior of Piezoresistive Smart Cement Contaminated with Oil Based Drilling Mud" OTC 2014, Paper No. OTC-25200-MS.
- 13. Vipulanandan, C. and Prashanth, P. (2013)." Impedance Spectroscopy Characterization of a Piezoresistive Structural Polymer Composite Bulk Sensor." Journal of Testing and Evaluation, Volume 41, No. 6, pp.1-13.
- Vipulanandan, C. and Garas, V. (2008) "Electrical Resistivity, Pulse Velocity and Compressive Properties of Carbon Fiber Reinforced Cement Mortar," Journal of Materials in Civil Engineering, Vol. 20, No. 2, pp. 93-101.
- 15. Vipulanandan, C. and Liu, J. (2005) "Polyurethane Based Grouts for Deep Off-Shore Pipe-in-Pipe Application", Proceedings, Pipelines 2005, ASCE, Houston, TX, pp. 216-227.
- Vipulanandan, C.,and Garas, V. (2006), "Piezoresistivity of Carbon Fiber Reinforced Cement Mortar", Proceedings, Engineering, Construction and Operations in Challenging Environments," Earth & Space 2006, Proceedings ASCE Aerospace Division, League City, TX, CD-ROM.
- 17. Vipulanandan, C. and Sett, K. (2004) "Development and Characterization of Piezoresistive Smart Structural Materials," Proceedings. Engineering, Construction and Operations in Challenging Environments, Earth & Space 2004, ASCE Aerospace Division, League City, TX, pp. 656-663.
- Welton, T.D., Todd, B.L., McMechan, D., 2010. "Methods for effecting controlled break in pH dependent foamed fracturing fluid". US Patent 7 662 756, assigned to Halliburton Energy Services, Inc., Duncan, OK, February 16, 2010.
- 19. Xu, B., Zhu, D., and Wang, L. (2011)." Experimental Evaluation of Guar-Fracture-Fluid Filter-Cake Behavior" SPE Production & Operations, pp.381-387.

Table 1. Test Methods and Mechanical Properties of Rock

Mechanical Property	Test Method	Value
Density, γ (gm/cm ³)	ASTM D 5195	2.45
Permeability, k (mD)	& CIGMAT Testing Standard 2011	6*10 ⁻¹⁰
Water Absorption (%)	ASTM D 6473	0.25
Porosity, n (%)	ASTM D 7263	0.55
Compressive Strength, σ_c (psi)	ASTM D7012	9000
Splitting Tensile Strength, σ_t (psi)	ASTM D3967	768
Modulus of Elasticity, E (psi)	ASTM D3148	450000

Table 2. Fracturing Fluid Mixes

Mix #	Water (%)	Sand (%)	Guar Gum (%)	Nano Silica (%)	Remark
1	90	9	1	0	Base Fluid
2	93	5	1	1	Effect of Nano Silica

Table 3. Model Parameters for Relationship between Shear Stress – Shear Strain Rate of Fracturing Fluids

Mix #	Temperature (°C)	τ _ο (Pa)	A	В	\mathbb{R}^2
	25	87.3	1.28	0.025	0.96
1	45	74.5	8.61	0.018	0.97
1	65	61.7	9.01	0.019	0.98
	85	55.6	10.55	0.018	0.99
	25	110.8	1.43	0.013	0.98
2	45	83.3	1.51	0.016	0.96
	65	70.6	1.67	0.016	0.96
	85	67.8	2.25	0.019	0.97

Table 4. Yield Stress and Viscosity Model Parameters for Fracturing Fluid Mixes

	Eqn. 3					
Rheological Properties	Mix	$\mathbf{Y}_{\mathbf{o}}$	A	В	\mathbb{R}^2	
Yield Stress	1	113.3	0.77	0.008	0.99	
(τ_o) , Pa	2	109.1	1.88	0.007	0.95	
Viscosity	1	1253.1	0.012	0.0008	0.99	
(at 170 s ⁻¹), cP	2	1505.3	0.009	0.0007	0.99	

Table 5. Fluid Loss – Time Model Parameters for Fracturing Fluid Modified with 1% Nano Silica for $85^{\circ}C \ge T \ge 25^{\circ}C$

Model Parameter	k	a	b	c	d	e	No. of Data	\mathbb{R}^2
A	1.21	-2.6	0.03	-0.014	0.055	-0.02	6	0.98
В	1.0	-1.5E-4	3.7E-4	1.57E-3	-2.53E-4	-8.4E-5	6	0.93

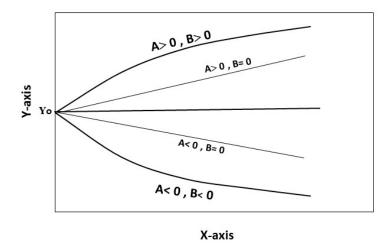


Figure 1. Modeling the Linear and Nonlinear Responses of the Fracturing Fluids

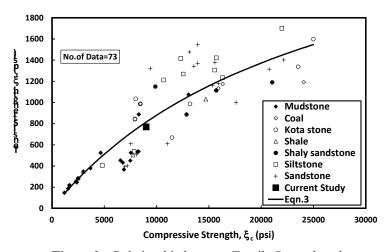


Figure 2. Relationship between Tensile Strength and Compressive Strength of the Rocks

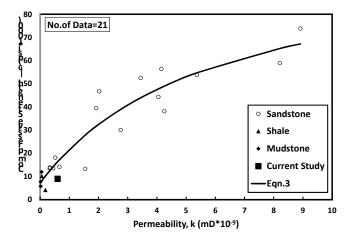
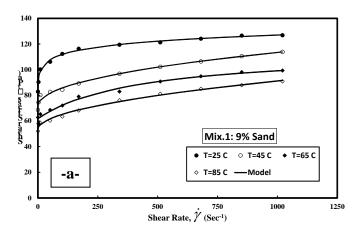


Figure 3. Relationship between Compressive Strength and Permeability of the Rocks



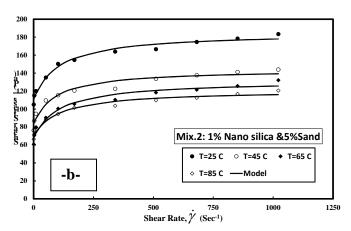


Figure 4. Measured and Predicted Shear Stress - Shear Strain Rate Relationship for Fracturing Fluids at Various Temperature: (a) 9% Sand (b) 1% Nano Silica and 5%Sand

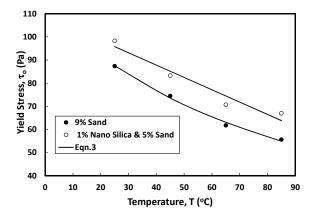


Figure 5. Measured and Predicted Yield Stress of Fracturing Fluids with Temperature

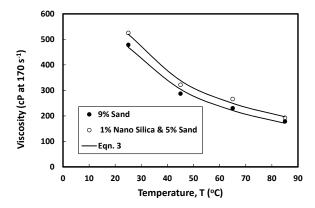


Figure 6. Measured and Predicated the Viscosity (at 170 sec⁻¹) of Fracturing Fluids with Varying Temperature

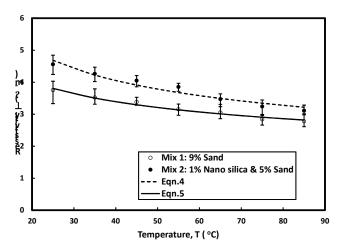
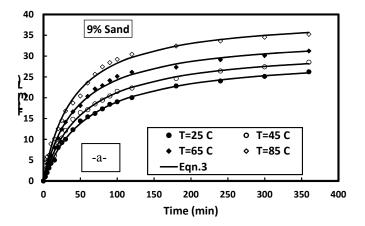


Figure 7. Measured and Predicated the Electrical Resistivity of Fracturing Fluids



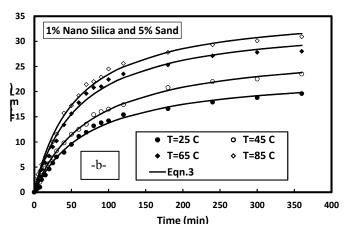


Figure 8. Measured and Predicted Kinetic of Fluid Loss in Fracturing Fluids with Temperature (a) 9% Sand (b) 1% Nano silica & 5% Sand

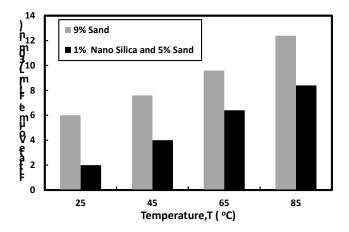
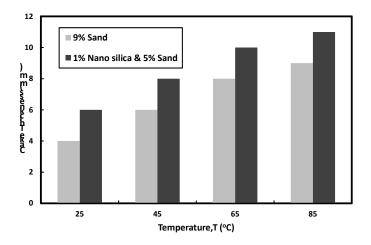
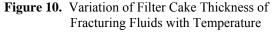


Figure 9. Variation of Fluid Loss Volume after 30 mints for Fracturing Fluids with Temperature





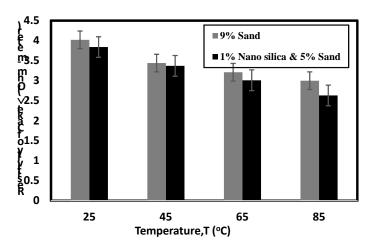


Figure 11. Variation of Resistivity of Filter Cake with Temperature for the Fracturing Fluids

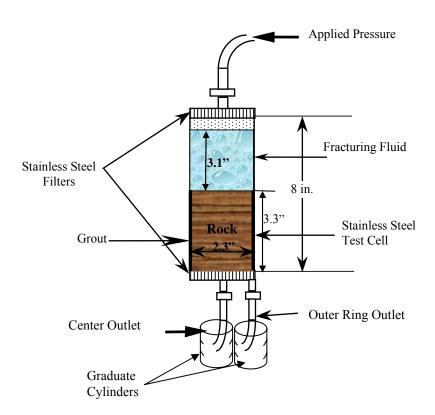


Figure 12. Double Ring HPHT Testing Device Used for the Sandstone Permeability Study

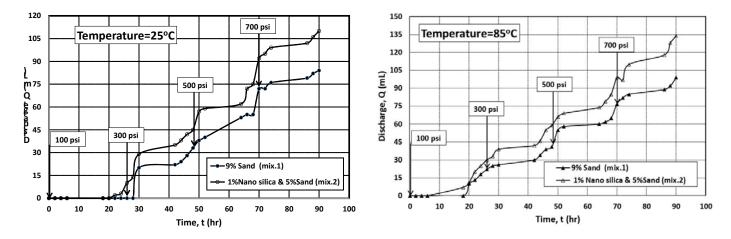


Figure 13. Fluid Discharge through Pre- Fractured Sandstone with Pressure and Temperature and Fracturing Fluids

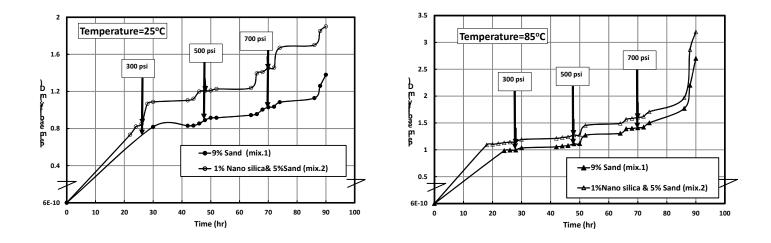


Figure 14. Variation of Permeability of the Pre- Fractured Rock with Pressure and Temperature and Fracturing Fluids