

## Monetizing Low Value Streams to High Value Emulsifiers: Performance Analysis and Field Application



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### Abstract

In today's era of low priced oil, operators and service providers sustain business by reducing their cost foot prints. A reduced cost foot print may involve lower pricing on existing or fit-for-purpose services and products, as well as compromised performance on the services and products. In this publication, we present high value primary and secondary emulsifiers for invert emulsion (oil-based) drilling fluids developed from a refinery side stream of the crude tall oil (CTO). We managed the composition variation challenge by setting precise specifications on this side stream and connecting aspects of product composition with desired performance in the drilling fluid application.

This side stream was derivatized under engineered reaction conditions to develop the present high-value primary and secondary emulsifiers without compromise in performance. Overall, detailed testing was performed to determine the emulsifier performance in different base oils (mineral oil and diesel) at different mud weights (12 to 16 ppg) characterized by rheology and HTHP fluid loss. The developed primary and secondary emulsifiers performed on par or outperformed the industry-available emulsifiers tested in this study.

The primary emulsifier demonstrated low pour points, low product viscosity and high flash points as compared to the industry standards. The field application of this primary emulsifier will be presented.

On the other hand, the secondary emulsifier provided a highly solids-tolerant, oil-based fluid as compared to the industry standards.

### Introduction

The rotary rig count today in the U.S. remains less than half of what it was in 2014<sup>1</sup>. The U.S. Energy Information Administration (EIA) reports that the rig and drilling costs make up 15 percent of the cost of drilling and completing the well<sup>2</sup>. Also, the drilling costs-per-unit depth has been declining continually since the highs of 2012<sup>2</sup>. These pressures cause operators to restructure and streamline operations to improve drilling efficiency. Every drilling budget, regardless of target formation, comprises the drilling rig, drill string, fluid system, casing and cement. Our focus in this paper is to improve the drilling efficiency of invert emulsion fluid (IEF) systems with the use of efficient emulsifiers. Emulsifiers are a

small portion (typically from 1 to 6 percent V/V) in the invert emulsion fluid but perform significant functions during the drilling process such as:

- Stabilize the invert emulsion.
- Maintain stable suspension.
- Maintain stable rheology.
- Oil-wet the drilled solids.
- Maintain controlled fluid loss to the formation.
- Maintain shale inhibition and stability.

During this research project, we developed:

- A high-value primary emulsifier (HVPE) that delivered a tight invert emulsion, thus it was more efficient at controlling high pressure high temperature (HPHT) fluid loss (FL) than the industry-available primary emulsifiers (PE). Also, the HVPE had lower pour points than the industry PE, thereby making it easier to handle even in colder temperatures.
- A highly solids-tolerant, high-value secondary emulsifier (HVSE) that imparted controlled (low) rheology to IEF in the presence of high concentrations of simulated drill solids. The advantages of this highly solids-tolerant secondary emulsifier are:
  - It can deliver low equivalent circulating density (ECD) fluids even at high concentrations of drill solids.
  - It can reduce the treatments needed to control high rheology and thereby improve the working longevity of the fluids.

The emulsifiers developed in this research were made from one of the variable side streams of the CTO distillation process. CTO is an acidulated product from sulfate black liquor skimming's which - on distillation - typically gives five product streams<sup>3</sup>:

1. Tall oil fatty acids (TOFA) that comprise ~30 - 35%
2. Tall oil rosin (TOR) that comprises ~30 - 40%
3. Distilled tall oil (DTO) that comprises ~5 - 10%
4. Tall oil heads that comprise ~5 - 10%
5. Tall oil pitch that comprises ~15 - 40%

Therefore, to provide these products streams, several variable side streams (VSSs) are produced. The current paper describes successful efforts to monetize the remaining fatty acid in one of these VSSs and manage variable properties to

provide products that perform consistently. The variability in an important chemical property (used as a quality control specification for TOFA) for this VSS is presented in Figure 1. Also, presented for comparison is the variability in the same chemical property for TOFA. The variability was determined by the subtracting individual batch values from the average of this chemical property.

The contents of this paper are arranged as follows:

- Development of high-value primary emulsifier (HVPE)
  - Comparison of HVPE with industry-available PE
  - Field data of HVPE-Based IEF used on a well
- Development of high-value secondary emulsifier (HVSE)
  - Data analysis and comparison with industry SE
  - Advantages of low (controlled) rheology in the presence of high volume of drill solids
- Performance of the HV emulsifier package in various fluid systems.
  - Performance in two mud weights – 16 ppg and 12 ppg
  - Performance of HV emulsifiers in a mineral base oil
  - Performance of HV emulsifiers in different fluid type – gilsonite-based FLA

## Materials and Methods

The fluids were mixed in stainless steel mixing cups on a five-spindle multimixer model 9B having a rotational speed of 11500 rpm with sine-wave impeller blade No. 9B29X. The fluids were aged in HTHP stainless steel aging cells and hot rolled in a model 5 roller oven from OFITE at the desired temperature for 16 h. The rheology of the fluids was determined on a standard OFITE oilfield viscometer as per API 13B-2. The rheology of the fluid was characterized in terms of the Bingham plastic viscosity (PV), Bingham yield point (YP) and gels minutes which were determined as per API 13B-2. The temperature was controlled with an electrically heated thermocup. In situations where the 600 rpm dial reading was not measurable due to instrument limitations, it was estimated by extrapolation with the Herschel-Bulkley rheological model. The emulsion stability (ES) was determined on an Electrical Stability Tester model 23E from FANN. HPHT fluid loss was determined on a 500 ml HPHT filter press assembly for duration of 30 minutes as per API 13B-2 recommendations.

The additives used to mix the fluids in this study were:

- Base oil: diesel and mineral oil
- Alkalinity control agent to activate the emulsifiers
- FLAs to maintain fluid loss control: polymeric FLA, amine-treated lignite and gilsonite FLA
- Viscosifiers to maintain viscosity and suspension: polymeric viscosifier, high quality organoclay and economical organoclay
- PE: HVPE and industry available PE-1, PE-2, PE-3 and PE-4
- SE: HVSE and industry available SE-1, SE-2 and SE-3
- Simulated drill solids

- $\text{CaCl}_2$  for water phase salinity (WPS)
- Weighting agent to adjust density of the fluid

## Results and Discussions

### Development of High-Value Primary Emulsifier

A survey of the on-shore drilling market in the U.S. showed that most of the IEFs use diesel fuel as the base oil. A 12 lb/gal (ppg) diesel fluid system was chosen as the screening formulation for the HVPE development, as shown in Table 1.

The HPHT fluid loss was considered the most significant parameter for the HVPE development. A lower HPHT fluid loss with no water in the filtrate was considered a sign of a good emulsifier with a fluid loss of less than 7 mL at 6 lb/barrel (ppb) PE concentration was preferred. In a laboratory experiment, 1 ppb refers to 1 g in 350 mL of the fluid. Since viscosifiers and FLA influence the HPHT fluid loss, concentrations of these were maintained at a necessary minimum in this screening formulation.

The screening formulation in Table 1 was used to optimize the reaction process parameters. Some of the reaction process parameters investigated for optimization were:

- Reaction temperature
- Reaction time
- Reactant concentrations and types
- Catalyst concentration
- Reactant specifications
- Product specifications

Table 2 provides the four batches of the HVPE made with the optimized reaction process. These HVPE batches were made with four different batches of the VSS. These batches of VSS had different numbers of standard deviation ( $\sigma$ ) distances from the average for the important chemical property as discussed previously. It was observed that these batches of the HVPE gave less than 7 mL with no water at 6 ppb concentration. In addition, the fluid loss at 5 ppb was low. The pour points of HVPE batches were all less than 0°C implying that these are good candidates for cold climate sites.

### Comparison of HVPE with Industry-Available PE

In Table 3, a comparison between the HVPE and Industry PE-1, PE-2, PE-3 and PE-4 in the screening formulation is shown. Analysis showed that:

- At 6 ppb, only the PE-1 with fluid loss of 6.6 mL came close to the fluid loss performance of HVPE
- At 5 ppb, the PE-1 gave fluid loss of 7.8 mL with 0.3 mL water as compared to 4.0 mL for the HVPE
- All the other industry PEs gave high HTHP fluid loss.

In Table 4, the product physical properties of the HVPE, PE-1, PE-2, PE-3 and PE-4 are compared. Analysis of the data shows that:

- HVPE had a low viscosity, thus it is easy to pour in general, particularly as compared to PE-1. The HVPE needs no dilution with solvents for application at the mud plant or rig site.

- The low pour points of the HVPE present it as a good candidate for use in cold climates, particularly compared to PE-1 and PE-2.
- The flash point of the HVPE was above 175°C, giving it an improved flammability profile as compared to the industry PE-2, PE-3 and PE-4.

#### **Field Data of HVPE-Based IEF Used on a Well**

As of April 2017, the HVPE was used on more than 50 wells for one Ingevity customer. We are presenting the data from its use on one lateral well in the U.S. in Table 5. The HVPE was used in this lateral well to drill to a measured depth of 22,550 feet. The dosage was about 3.2 ppb of the HVPE for this 9.7 ppg mud. It had overall stable, consistent rheology and a controlled fluid loss.

#### **Development of High-Value Secondary Emulsifier (HVSE)**

SEs are used in both conventional and polymeric fluid systems, alone or with PEs. The conventional and polymer fluids may differ in the viscosifier and FLA additive packages used in these systems.

- A conventional fluid system typically uses an amine-treated lignite as the FLA and an organoclay (a fatty quaternary amine treated bentonite) as the viscosifier.
- A polymeric fluid system typically uses a polymeric fluid loss additive and an organic viscosifier like dimer-trimer fatty acid, a fatty acid or a fatty acid-amine derivative.

Thus, these two fluid system types were used to optimize the reaction process. Similar parameters were considered as with the HVPE for the development of the HVSE. The conventional and polymer fluid screening formulations used in this optimization are given in Table 6 and Table 7 respectively. In the conventional formulation, industry PE-2 was added with the SE to form the emulsion. On the other hand, SE was used as the only emulsifier in the polymer formulation.

The SE is usually expected to oil-wet drill solids. These drill solids, if not oil-wet, can cause an increase in the HPHT fluid loss at lower concentration and can lead to emulsion destabilization at higher concentrations in the IEF. Therefore, the development of the HVSE was conducted by optimizing the reaction process to reduce the impact of adding 100 ppb of simulated drill solids to the conventional and polymer systems. (The 100 ppb drill solids conventional and polymer formulations are given in the Appendix in Table 15 and Table 16, respectively).

The main parameter used to quantify performance for the development of the HVSE was the HTHP fluid loss. The goal was to optimize for the lowest fluid loss possible at

- 4.5 ppb (3.0 ppb at 100 percent activity) SE concentration for the conventional fluid system in the presence and absence of 100 ppb simulated drill solids.
- 13 ppb (8.7 ppb at 100 percent activity) SE concentration for polymer fluid system

- 16 ppb (10.7 ppb at 100 percent activity) SE concentration for the polymer fluid system with 100 ppb simulated drill solids.

Thus, the development of the HVSE was guided primarily by the HTHP results of these four fluid formulations: 16.4 ppg conventional fluid; 16.4 ppg conventional fluid with 100 ppb drill solids; 14.57 ppg polymeric fluid; and 14.57 ppg polymeric fluid with 100 ppb drilled solids.

Although we targeted development around the HTHP fluids loss for the HVSE development, we later found that the fluid rheology effects of the HVSE were also significant.

#### **Data Analysis**

##### **IEF in Absence or Low Concentration of Simulated Drill Solids**

After the reaction process optimization, several batches of the HVSE were made with the optimized reaction process. Data of three batches of the HVSE tested in the conventional and polymer fluid screening formulations are given in Table 8 and Table 9, respectively. For comparison, the data of industry SE-1 and SE-2 in the conventional and polymer fluids are also given in Table 8 and Table 9. Analysis of the data yields the following conclusions:

- The gels at 10 minutes and the Bingham YP of the HVSE-based conventional and polymer fluid systems were lower than the industry SE-1 and SE-2.
- The ES value of the HVSE-based conventional fluid system was comparable to the industry standards. In the polymer fluid system, the ES of the HVSE-based IEF was comparable to SE-1 but higher than the SE-2-based IEF.

The HTHP fluid loss of the

- HVSE-based conventional fluid was comparable to SE-1 but lower than the SE-2-based conventional fluid.
- HVSE-based polymer fluid was comparable to industry SE-1 and SE-2-based polymer fluid,
  - the HVSE-based polymer fluid did not have water in the filtrate.
  - The SE-1 and SE-2-based polymer fluid showed presence of water in the filtrate. The presence of water in the filtrate may be considered signs of emulsion instability.

On analyzing the dial readings for shear rates from 600 rpm to 3 rpm (Table 17 and Table 18 in the Appendix) it is evident that the HVSE-based IEF has lower rheology than the industry SE-1 and SE-2 based IEF in both conventional and polymer fluid systems.

##### **IEF in Presence of 100 ppb Simulated Drill Solids**

Data of the three HVSE batches and industry SE-1 and SE-2 tested in conventional and polymer fluids in presence of 100 ppb simulated drill solids is given in Table 10 and Table 11.

In the conventional fluid systems that were loaded with 100 ppb simulated drill solids, the 600 rpm dial reading (DR) at 120°F was unmeasurable due to the limitation of the instrument. Therefore, the 600 rpm dial reading was estimated

by extrapolation with the Herschel-Bulkley model.

Anticipating a similar situation with the polymer fluid with 100 ppb simulated drill solids, the rheology of these polymer fluids was determined at 150°F.

In the conventional fluid (Table 10):

- When comparing the Bingham PV, the HVSE-based conventional fluid on average was marginally higher than industry SE-1 and SE-2.
- When comparing the Bingham YP, the HVSE-based conventional fluid was on average 17 to 23 percent lower than the industry SE-1 and SE-2-based conventional fluids.
- Gels at 10 minutes for the HVSE-based conventional fluids were on an average 20 to 25 percent lower than the industry SE-1 and SE-2-based conventional fluids.
- The ES value of the HVSE-based conventional fluid system was approximately 100 V higher than the industry SE-1 and SE-2.
- The fluid loss value of the HVSE-based conventional fluid systems was lower than the industry SE-1 and SE-2-based conventional fluids. Though one of the HVSE batches gave a high fluid loss of 21 mL with 2 mL water, the other batches gave lower fluid loss values with no water in the filtrate. The industry SE-1 and SE-2 gave high fluid losses at 22.4 and 24.0 mL with 2.0 and 4.0 mL, respectively. The presence of water in the filtrate may be considered signs of emulsion instability.

On analyzing the dial readings from 600 rpm to 3 rpm in the conventional fluid system, the following were found (Table 19 in Appendix):

- From 600-100 rpm, HVSE on average gave marginally lower dial readings compared to the industry SE-1 and SE-2.
- From 6 to 3 rpm, the HVSE on average gave 35 to 40 percent lower dial readings as compared to the industry SE-1 and SE-2.

Regarding the polymer fluid in Table 11:

- On comparing the Bingham PV, the HVSE-based polymer fluid was roughly 30 percent and 20 percent lower than industry SE-1 and SE-2-based polymer fluids, respectively.
- On comparing the Bingham YP, the HVSE-based polymer fluids were roughly 70 percent and 60 percent lower than the industry SE-1 and SE-2-based polymer fluids respectively.
- Gels at 10 minutes for the HVSE-based polymer fluid were roughly 66 percent and 40 percent lower than the industry SE-1 and SE-2-based polymer fluids, respectively.
- The ES of the HVSE-based polymer fluid was roughly 150 V lower than the industry SE-1 but was comparable to industry SE-2-based polymer fluids.
- The fluid loss of the HVSE in the polymer fluid systems was on an average (FL of 9.5 mL) higher compared to industry SE-1 based polymer fluids (FL of 7.2 mL) and lower than the industry SE-2-based polymer fluid. All the

HVSE batches and the industry SE-1 and SE-2 gave water in the filtrate.

On analyzing the dial readings from 600 rpm to 3 rpm in the polymer fluid system (Table 20 in Appendix) presented in Figure 2, HVSE-based polymer fluids gave substantially lower dial readings from 600 to 3 rpm as compared to the industry SE-1 and SE-2-based polymer fluids. Further:

- Between 600 to 100 rpm, dial readings of the HVSE-based polymer fluids were 42 to 63 percent lower compared to industry SE-1-based polymer fluids, and 28 to 48 percent lower compared to the SE-2-based polymer fluid.
- Between 6 to 3 rpm, dial readings of the HVSE-based polymer fluids were 72 to 83 percent lower than industry SE-1-based polymer fluids, and 44 to 65 percent lower than industry SE-2-based polymer fluids.

### ***Advantages of Low (Controlled) Rheology in the Presence of High Volume of Drill Solids***

During the drilling process, drill solids tend to degrade into progressively smaller particles. These fine particles ultimately remain unfiltered by the solids control equipment due to their small size. As drilling progresses, these finer particles attain colloidal size and steadily increase in their concentration in the drilling fluid. These colloidal fines and their concentration can negatively impact the performance of the IEF, one of the main symptoms being an uncontrolled increase in rheology. This increase in rheology tends to increase the equivalent circulating density (ECD) of the fluid. The ECD is the effective density of the circulating fluid in the wellbore resulting from the sum of the density of the fluid and pressure drop across the annulus. The increased rheology of the drilling fluid results in a high-pressure drop across the annulus, thereby increasing the ECD. The ECD is an important parameter in avoiding kicks and losses of the fluid, particularly in wells that have a narrow window between the fracture gradient and pore-pressure gradient. Thus, it is observed that the HVSE delivers controlled, low rheology as compared to industry SE-1 and SE-2 in fluid systems loaded with high volumes of low gravity solids.

With increases in colloidal fines in the IEF, the fluid needs treatment to reduce negative impacts, which often involves a least one of the below:

- Adding a thinner to lower the rheology
- Adding wetting agents to maintain the water wettability of the colloidal fines
- Adding fresh IEF to dilute the overall drilling fluid to reduce the overall concentration of the low gravity solids (LGS)
- Performing high speed centrifuge operations to remove the LGS solids
- Adding other additives to maintain a workable fluid.

However, at times these treatments may not work and the IEF must be discarded and fresh IEF must be made. Adding the HVSE to the IEF may reduce the maintenance/treatments of these fluids by maintaining a controlled (low) rheology

profile and fluid loss in the IEF. This improvement may, in turn, increase the overall working longevity of the IEF.

### **Performance of the HV Emulsifier Package in Various Fluid Systems**

#### **Performance in Two Mud Weights – 16 ppg and 12 ppg**

HVPE and SE emulsifiers were then tested together in a conventional fluid system. The conventional fluid system was formulated with diesel as the base oil and industry-available amine-treated lignite and a high-quality organoclay. Laboratory trials were conducted to determine the optimum concentration of the additives and optimum ratio of the emulsifiers for these formulations. The emulsifier ratio of 3:1 for the PE:SE was determined as optimum for both the 16 ppg and 12 ppg fluids.

In the 16 ppg fluid, total emulsifier concentration of 12 ppb was determined as appropriate whereas in the 12 ppg fluid, total emulsifier concentration of 8 ppb was determined appropriate.

In the 16 ppg formulation (Table 12) we also tested the industry emulsifier package of PE-1 and SE-3. On comparison, the HV emulsifiers gave overall low rheology compared to the industry emulsifier package of PE-1 and SE-3. The PV, YP and gels at 10 minutes were all lower for the HV emulsifier fluid. The HTHP fluid loss and the ES for both fluid systems were similar with 4.2 mL and 835 V for HV formulation, as compared to 3.6 mL and 910 V for the PE-1 and SE-3 formulation.

The additives used in the 12 ppg system were the same as the 16 ppg fluids systems, but the optimum concentrations of the additives used in the 12 ppg system was determined to be different (Table 12, right hand side of column). A stable 12 ppg fluid was obtained with the HV package, with controlled HPHT fluid loss of only 3.6 mL and ES of 820 V.

Thus, stable 16 ppg and 12 ppg stable fluids were obtained with the HV emulsifier package.

#### **Performance of HV Emulsifiers in a Mineral Base Oil**

Then the performance of the HV emulsifier was tested in a commonly used mineral oil-based conventional fluid system as given in Table 13. The same additives and their same concentrations were used to mix the mineral oil-based fluid as for the diesel (as given in Table 12).

- The mineral oil-based fluid gave low higher-end rheology (600 to 300 rpm dial readings) and high lower-end rheology (6 to 3 rpm dial readings) as compared to the diesel-based fluid system.
- The gels at 10 minutes were substantially high at 20 units for mineral oil-based fluid as compared to 3 units for the diesel-based fluid.
- The ES of the mineral oil-based fluid was higher at 1020 V compared to 835 V for the diesel-based fluid.
- The HPHT fluid loss at 350 °F of the mineral fluid system was controlled at 6.0 mL (Table 13).

Thus, the HV emulsifier package gave stable 16 ppg fluids in both diesel and mineral oil.

### **Performance of HV Emulsifiers in Different Fluid Type – Gilsonite-Based FLA**

Then it was determined to study the HV emulsifier package in the conventional fluid system, but with an economical organoclay and gilsonite-type fluid loss additive as given in Table 14. The fluid parameters were same as those of the previous section on performance in different mud weights. The concentration of all the functional additives in the gilsonite-based fluid systems for 16 ppg and 12 ppg were like the previous diesel-based fluid except that in the 16 ppg fluid, the gilsonite-based FLA was used at 8 ppb as compared to 6 ppb for the amine-treated lignite.

Both the 16 ppg and 12 ppg gilsonite-based fluid systems were stable and had reasonable rheology. The gilsonite-based fluids (Table 14) had lower ES values and higher HTHP fluid losses as compared to the amine-treated lignite-based fluids (Table 12).

Thus, the HV emulsifier package gave stable 16 ppg and 12 ppg fluids in gilsonite and economical organoclay type, diesel-based fluids.

### **Conclusions**

1. Two efficient high-value primary and secondary emulsifiers were developed with the use of a variable side stream from the CTO distillation process.
2. The HVPE had better fluid loss control as compared to the industry-available PE used in this study.
3. The HVSE demonstrated high tolerance to drilled solids by giving controlled low rheology as compared to the industry SE in the IEF loaded with 100 ppb of drill solids.
  - a. The HVSE may be used for low ECD requirements at high concentrations of drilled solids.
  - b. The HVSE may reduce the need for treatments for high concentrations of drill solids in the IEF and improve the working longevity of the IEF.
4. The HVSE and HVPE emulsifier package:
  - a. Performed comparably to the industry emulsifier packages in conventional fluids systems.
  - b. Delivered stable 16 ppg and 12 ppg conventional fluids with controlled fluid loss and stable rheology.
  - c. Delivered stable 16 ppg conventional fluids in diesel and mineral oil with controlled fluid loss and stable rheology.
  - d. Delivered stable 16 ppg conventional fluids with gilsonite-based FLA and economical organoclay package.

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## Nomenclature

<i>API</i>	= American Petroleum Institute
<i>CTO</i>	= Crude Tall Oil
<i>°C</i>	= Degree Celsius
<i>cP</i>	= Centipoise
<i>DND</i>	= Did Not Do
<i>DNM</i>	= Did Not Measure
<i>D.R.</i>	= Dial Reading
<i>DTO</i>	= Distilled Tall Oil
<i>ECD</i>	= Equivalent Circulating density in ppg
<i>EIA</i>	= Energy Information Administration
<i>ES</i>	= Emulsion Stability
<i>°F</i>	= Degree Fahrenheit
<i>FL</i>	= Fluid loss
<i>FLA</i>	= Fluid Loss Additive
<i>g</i>	= grams
<i>h</i>	= hours
<i>HPHT</i>	= High Pressure High Temperature
<i>HV</i>	= High Value
<i>HVPE</i>	= High Value Primary Emulsifier
<i>HVSE</i>	= High Value Secondary Emulsifier
<i>IEF</i>	= Invert Emulsion Fluids
<i>LGS</i>	= Low Gravity Solids
<i>mL</i>	= milliliter
<i>mins</i>	= Minutes
<i>OWR</i>	= Oil Water Ratio
<i>PE</i>	= Primary Emulsifier
<i>ppb</i>	= lb/bbl
<i>ppg</i>	= lb/gal
<i>PV</i>	= Plastic Viscosity
<i>rpm</i>	= Revolutions per minute
<i>SE</i>	= Secondary Emulsifier
<i>TOFA</i>	= Tall Oil Fatty Acids
<i>TOR</i>	= Tall Oil Rosins
<i>TVD</i>	= True Vertical Depth
<i>V/V</i>	= Volume/Volume
<i>VSS</i>	= Variable Side Stream
<i>WPS</i>	= Water Phase Salinity
<i>w/w</i>	= Weight/Weight
<i>YP</i>	= Yield Point
$\sigma$	= Standard Deviation

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## Figures

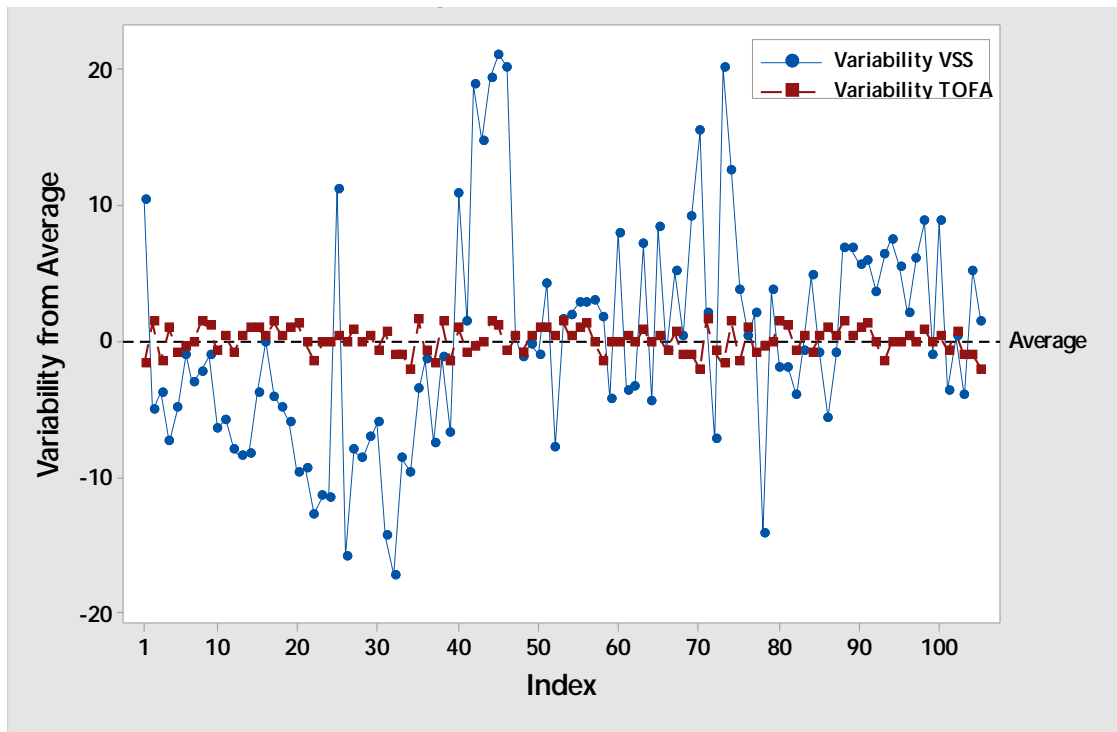


Figure 1 : Variability in the chemical property for VSS and TOFA for a sample set

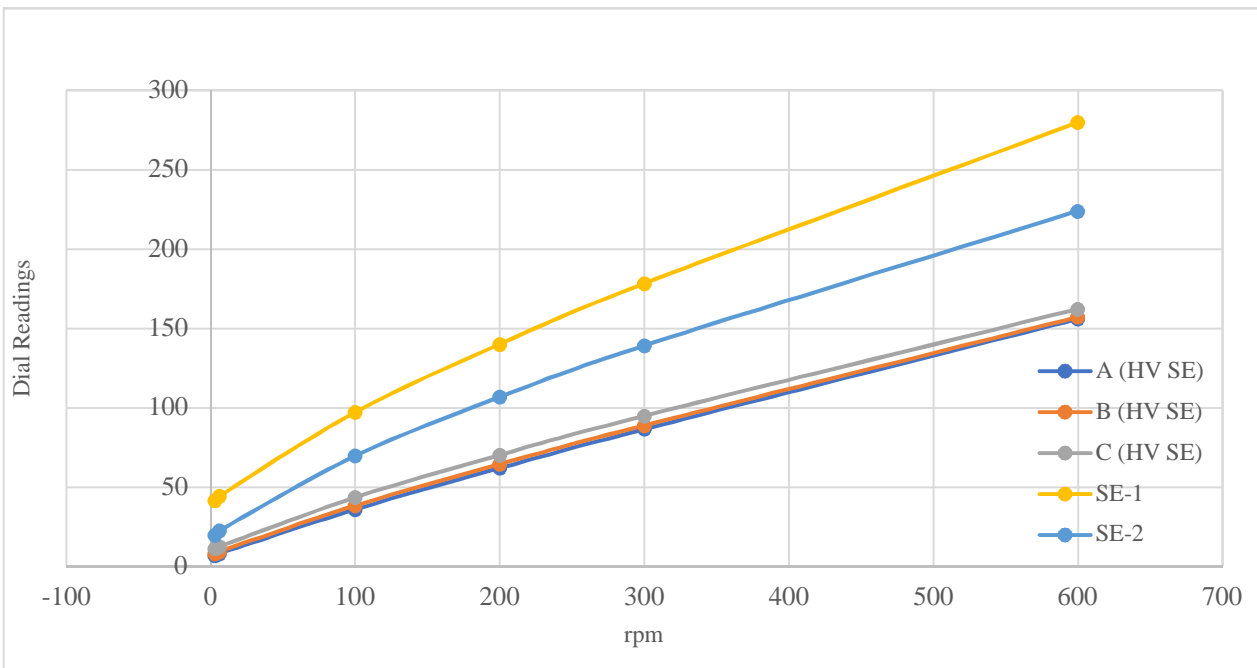


Figure 2: Rheogram at 150°F of HVSE compared with SE-1 and SE-2 in 14.57 ppq polymer fluid with 100 ppb simulated drill solids

## Tables

Components	Specific Gravity	Mass (g)**	Mixing (min)
Low sulfur diesel	0.86	185.5	0
Primary emulsifier	0.99	6.0	5
Alkalinity control agent	2.24	3.0	10
CaCl <sub>2</sub> Brine (WPS = 25% w/w)	1.24	89.1	10
Amine treated lignite	1.80	4.0	10
High quality organoclay	1.70	4.0	10
Weighting agent	4.20	212.0	20
Total		503.6 grams	65 mins
OWR	75:25		

\*\* refers to grams in 350 mL volume of the fluid which is equivalent to ppb.

HVPE batches	VSS, chemical property's number of $\sigma$ distance from average	Pour Point (°C)	FL at 300°F and at 6 ppb, mL	FL at 300°F and at 5 ppb, mL
1	1.25	-12	2.8	7.0
2	-0.66	-12	4.8	7.6
3	3.29	-18	4.0	4.0
4	-0.01	-21	3.2	4.8

	FL at 300°F and at 6 ppb		FL at 300°F and at 5 ppb	
	Total loss (mL)	water (mL)	Total loss (mL)	water (mL)
Industry PE – 1	6.6	-	7.8	0.3
Industry PE – 2	32.4	1.4	DND	DND
Industry PE – 3	36.0	-	DND	DND
Industry PE – 4	16.0	-	DND	DND
HVPE batch # 3	4.0	-	4.0	-

DND – Did not do.

Property	HVPE	PE-1	PE-2	PE-3	PE-4
Viscosity at 25°C (cP)	150 – 1200	5000 – 10,000	≈ 500	90-130	13.5
Pour Point (°C)	-9 to -27	12 to 16	3.5	<-20	<-20
Flash Point (°C)	>175	>175	156	82	94

Table 5: Field data of the use of the HVPE on a well

Depth/TVD, ft	6380 / 6325	13718 / 7237	17218 / 7217	20550 / 7205	22016 / 7191	22016 / 7191
	Drilling Ahead.	Drilling Ahead.	Drilling ahead.	Drilling Ahead.	L/D DP	RUNNING CASING
Mud Weight, ppg	9.6 @ 101°F	9.65 @ 141°F	9.75 @ 123°F	9.75 @ 132°F	9.8 @ 126°F	9.75 @ 100°F
Rheology temperature, °F	150	150	150	150	150	150
600 rpm, D.R.	61	60	60	76	75	75
300 rpm, D.R.	38	36	35	45	44	44
200 rpm, D.R.	30	27	26	35	32	32
100 rpm, D.R.	20	17	17	22	20	20
6 rpm, D.R.	7	5	5	6	6	6
3 rpm, D.R.	6	4	4	5	4	4
PV, cP	23	24	25	31	31	31
YP, lb/100ft <sup>2</sup>	15	12	10	14	13	13
Gel 10 sec	10	9	9	8	7	7
Gel 10 min	17	20	21	24	22	22
Gel 30 min	20	24	24	32	25	25
HPHT FL temperature, °F	250	250	250	250	250	250
HPHT FL, mL/30min	6.8	5	5	5.4	6	6.2
Cake HTHP, 1/32"	2.6	2	2	2.1	2.2	2.2
Oil water ratio	82/18	86/14	85/15	87/13	88/12	88/12
E-Stability	671	881	750	732	655	646
Water phase salinity, mg/L	207220	227019	218893	208434	227019	218893

Table 6: 16.4 ppg conventional fluid formulation for HVSE, hot rolled at 150°F for 16 h

	S.G.	Mass (g)	Time (min)
Low sulfur diesel	0.86	144.5	
PE-2	0.93	6.0	1
Alkalinity control agent	2.24	3.0	1
Amine treated lignite	1.80	6.0	1
SE – 66.7% (w/w) active	0.95	4.5	1
CaCl <sub>2</sub> (WPS = 33.5% w/w)	1.32	93.6	10
High quality organoclay	1.70	2.0	15
Weighting agent	4.20	470.0	10
Total	16.4 ppg	729.6 g	39 mins
OWR	70:30		

Table 7: 14.57 ppg polymeric fluid formulation, hot rolled at 325°F for 16 h

Product	S.G.	Mass (g)	Time (min)
Mineral oil	0.80	134.6	
SE – 66.7% (w/w) activity	0.95	13.0	1
Alkalinity control agent	2.24	3.0	1
Polymeric FLA	1.03	3.0	10
CaCl <sub>2</sub> (WPS = 30% w/w.)	1.29	92.6	10
Polymeric viscosifier	0.96	3.0	5
Simulated drill solids	2.50	20.00	5
Weighting agent	4.20	341.9	10
Total	14.57 ppg	611.3 g	42 mins
OWR	70:30		

**Table 8: Performance of HVSE compared with industry SE-1 and SE-2 in conventional fluid.  
HPHT fluid loss at 350°F. Rheology and ES determined at 120°F**

Product	VSS, chemical property, number of $\sigma$ distance from average	Gel 10 min (D.R.)	ES (V)	PV (cP)	YP (lb/100 ft <sup>2</sup> )	FL at 350°F (mL)
A	-1.93	4.0	489	33.2	8.3	7.6
B	-0.70	4.0	480	37.3	6.7	8.0
C	-0.05	4.0	441	38.1	4.7	6.8
SE-1		7.0	428	33.8	16.9	9.8
SE-2		6.0	500	35.8	12.6	14.0

**Table 9: Performance of HVSE compared with industry SE-1 and SE-2 in polymer fluid.  
HPHT fluid loss at 350°F. Rheology and ES determined at 120°F**

Product	VSS, chemical property, number of $\sigma$ distance from average	Gel 10 min (D.R.)	ES (V)	PV (cP)	YP (lb/100 ft <sup>2</sup> )	FL at 350°F (mL)	Water at 350°F (mL)
A	-1.93	2.0	378	31.6	0.8	6.0	-
B	-0.70	3.0	347	24.8	4.2	6.8	-
C	-0.05	2.0	255	28.6	2.0	7.6	-
SE-1		6.0	393	34.7	7.7	6.8	1.6
SE-2		13.0	163	47.6	19.2	3.2	2.0

**Table 10: Performance of HVSE compared with industry SE-1 and SE-2 in 100 ppb simulated drill solids in conventional fluid. HPHT fluid loss at 350°F. Rheology and ES determined at 120°F**

Product	VSS, chemical property, number of $\sigma$ distance from average	Gel 10 min (D.R.)	ES (V)	PV (cP)	YP (lb/100 ft <sup>2</sup> )	FL at 350°F (mL)	Water at 350°F (mL)
A	-1.93	27.0	235	183.5	64.5	21.0	2.0
B	-0.70	27.0	256	189.8	57.0	14.0	-
C	-0.05	29.0	245	201.7	65.2	9.2	-
SE-1		37.0	138	186.9	80.3	22.4	2.0
SE-2		36.0	101	183.8	75.7	24.0	4.0

**Table 11: Performance of HVSE compared with industry SE-1 and SE-2 in 100 ppb simulated drill solids in polymer fluid. HPHT fluid loss at 350°F. Rheology and ES determined at 150°F**

Product	VSS, chemical property, number of $\sigma$ distance from average	Gel 10 min (D.R.)	ES (V)	PV (cP)	YP (lb/100 ft <sup>2</sup> )	FL at 350°F (mL)	Water at 350°F (mL)
A	-1.93	15.0	141	69.4	17.1	8.0	2.0
B	-0.70	18.0	147	68.2	20.7	8.8	2.4
C	-0.05	21.0	144	67.3	27.6	11.6	DNM
SE-1		53.0	300	101.5	76.8	7.2	2.4
SE-2		30.0	204	84.8	54.4	12.8	4.0

DNM – Did not measure

Table 12: HV emulsifiers package of this study in two different mud weights – 16 and 12 ppg and compared with industry emulsifiers PE-1 and SE-3 package at 16 ppg

		HVPE and HVSE	PE-1 and SE-3	HVPE and HVSE	Mix Time
	S.G	Mass (g)	Mass (g)	Mass (g)	mins
Low sulfur diesel	0.86	163.3	163.3	179.5	0
PE	0.99	9.0	9.0	6.0	1
Alkalinity control agent	2.24	3.0	3.0	3.0	1
Amine treated lignite	1.8	6.0	6.0	6.0	1
SE – 66.7% (w/w) active	0.95	3.0	3.0	2.0	1
CaCl <sub>2</sub> (WPS = 30% w/w)	1.29	43.5	43.5	-	3
CaCl <sub>2</sub> (WPS = 25% w/w)	1.24	-	-	86.3	
High quality organoclay	1.7	2.0	2.0	8.0	3
Simulated drill solids	2.5	20.0	20.0	20.0	5
Weighting agents	4.2	421.2	421.2	192.6	15
	Total	671.1 g	671.1 g	503.3 g	30 mins
	OWR	0.85	0.85	0.75	
	Hot roll temperature (time)	350°F (16h)	350°F (16h)	325°F (16h)	
	Mud weight, ppg	16	16	12	
<b>Rheology @ 120°F</b>					
	600 rpm (D.R.)	80.1	102.9	88.1	
	300 rpm (D.R.)	41.3	55.8	47.6	
	200 rpm (D.R.)	28.7	39.7	33.6	
	100 rpm (D.R.)	15.7	23	19.6	
	6 rpm (D.R.)	1.8	3.4	7.6	
	3 rpm (D.R.)	1.5	2.9	7.3	
	PV (cP)	38.8	47.1	40.5	
	YP(lb/100ft <sup>2</sup> )	2.5	8.7	7.1	
	10" Gel (D.R.)	1.5	3.0	9.0	
	10' Gel (D.R.)	3.0	9.0	21.0	
	ES (V) at 120°F	835	910	820	
	HTHP FL (mL)	4.2 at 350°F	3.6 at 350°F	3.6 at 325°F	

	S.G	Mass (g)	Mass (g)	mins
Low sulfur diesel	0.86	163.3	-	0
Mineral oil	0.80	-	147.4	
HVPE	0.99	9.0	9.0	1
Alkalinity control agent	2.24	3.0	3.0	1
Amine treated lignite	1.8	6.0	6.0	1
HVSE – 66.7% (w/w) activity	0.95	3.0	3.0	1
CaCl <sub>2</sub> (WPS = 30% w/w)	1.29	43.5	42.7	3
High quality organoclay	1.7	2.0	2.0	3
Simulated drill solids	2.5	20.0	20.0	5
Weighting agents	4.2	421.2	438.0	15
	Total	671.1 g	671.1 g	30 mins
	OWR	0.85	0.85	
	Hot roll temperature (time)	350°F (16h)	350°F (16h)	
	Mud weight, ppg	16	16	
<b>Rheology @ 120°F</b>				
	600 rpm (D.R.)	80.1	68.5	
	300 rpm (D.R.)	41.3	35.1	
	200 rpm (D.R.)	28.7	24.4	
	100 rpm (D.R.)	15.7	13.7	
	6 rpm (D.R.)	1.8	2.6	
	3 rpm (D.R.)	1.5	2.6	
	PV (cP)	38.8	33.4	
	YP(lb/100ft <sup>2</sup> )	2.5	1.7	
	10" Gel (D.R.)	1.5	3.0	
	10' Gel (D.R.)	3.0	19.0	
	ES (V) at 120°F	835	1020	
	HPHT FL (mL) at 350°F	4.2	6.0	

Table 14: HV emulsifiers of this study tested in an economical organoclay and gilsonite based conventional fluid

	S.G	Mass (g)	Mass (g)	mins
Low sulfur diesel	0.86	159.8	177.5	0
Economical organoclay	1.70	2.0	8.0	2
Gilsonite FLA	1.06	8.0	6.0	2
HVPE	0.99	9.0	6.0	2
HVSE – 66.7% (w/w) active	0.95	3.0	2.0	2
CaCl <sub>2</sub> (WPS = 30% w/w)	1.29	42.6	-	5
CaCl <sub>2</sub> (WPS = 25% w/w)	1.24	-	85.3	
Alkalinity control agent	2.24	3.0	3.0	2
Simulated drill solids	2.50	20.0	20.0	5
Weighting agent	4.20	423.7	195.5	10
	Total	671.1 g	503.3 g	30 mins
	Mud weight, ppg	16	12	
	OWR	0.85	0.75	
	Hot roll temperature (time)	350°F (16h)	325°F (16h)	
<b>Rheology @ 120°F</b>				
	600 rpm (D.R.)	111.4	72.9	
	300 rpm (D.R.)	59.3	38.5	
	200 rpm (D.R.)	41.9	26.6	
	100 rpm (D.R.)	23.8	13.9	
	6 rpm (D.R.)	3.4	1.3	
	3 rpm (D.R.)	2.7	1.0	
	PV (cP)	52.1	34.4	
	YP(lb/100ft <sup>2</sup> )	7.3	4.1	
	10" Gel (D.R.)	2.0	0.0	
	10' Gel (D.R.)	7.0	6.0	
	ES (V) at 120°F	630	451	
	HPHT FL (mL)	11.6 at 350°F	5.6 at 325°F	

## Appendix

Table 15: 16.4 ppg conventional fluid formulation with 100 ppb simulated drill solids for HVSE, hot rolled at 150°F for 16 h

Products	S.G.	Mass (g)	Time (min)
Low sulfur diesel	0.86	126.9	
PE-2	0.93	6.0	1
Alkalinity control agent	2.24	3.0	1
Amine treated lignite	1.80	6.0	1
SE – 66.7% (w/w) active	0.95	4.5	1
CaCl <sub>2</sub> (WPS = 33.5% w/w)	1.32	83.5	10
High quality organoclay	1.70	2.0	5
Simulated drill solids	2.50	100.0	10
Weighting agent	4.20	355.9	10
	Total	16.4 ppg	39 mins
	OWR	70:30	

Table 16: 14.57 ppg polymeric fluid formulation with 100 ppb simulated drill solids, hot rolled at 325°F for 16 h

Product	S.G.	Mass (g)	Time(min)
Mineral oil	0.80	125.2	
SE – 66.7% (w/w) active	0.95	16.0	1
Alkalinity control agent	2.24	3.0	1
Polymeric FLA	1.03	3.0	10
CaCl <sub>2</sub> (WPS = 30% w/w)	1.29	86.2	10
Polymeric viscosifier	0.96	3.0	5
Simulated drill solids	2.50	100.0	5
Weighting agent	4.20	274.8	10
Total	14.57 ppg	611.1 g	42 mins
OWR	70:30		

Table 17: Rheology of HVSE compared with industry SE-1 and SE-2 at 120°F in 16.4 ppg conventional fluid

Product	600 rpm (D.R.)	300 rpm (D.R.)	200 rpm (D.R.)	100 rpm (D.R.)	6 rpm (D.R.)	3 rpm (D.R.)
A	74.7	41.5	29.7	17.0	3.3	2.8
B	81.2	43.9	31.0	17.6	3.8	3.3
C	80.9	42.8	29.8	16.6	3.6	3.2
SE-1	84.5	50.7	38.1	24.1	7.4	6.6
SE-2	84.1	48.3	35.6	21.9	7.0	6.3

Table 18: Rheology of HVSE compared with Industry SE-1 and SE-2 at 120°F in 14.57 ppg polymer fluid

Product	600 rpm (D.R.)	300 rpm (D.R.)	200 rpm (D.R.)	100 rpm (D.R.)	6 rpm (D.R.)	3 rpm (D.R.)
A	64.0	32.4	22.0	11.6	2.0	1.7
B	53.9	29.1	20.2	10.7	0.3	0.0
C	59.3	30.7	20.9	10.9	1.0	0.7
SE-1	77.1	42.4	30.3	17.6	4.6	4.1
SE-2	114.3	66.7	49.2	30.0	7.5	6.5

Table 19: Rheology of HVSE compared with industry SE-1 and SE-2 at 120°F in 16.4 ppg conventional fluid with 100 ppb simulated drilled solids

Product	600 rpm (D.R.)	300 rpm (D.R.)	200 rpm (D.R.)	100 rpm (D.R.)	6 rpm (D.R.)	3 rpm (D.R.)
A	431.5	248.0	180.1	105.3	17.0	12.7
B	436.6	246.8	177.5	102.2	15.9	11.9
C	468.6	266.9	192.8	111.8	17.6	13.2
SE-1	454.1	267.2	197.4	119.8	26.1	21.4
SE-2	443.3	259.5	191.3	116.1	26.8	22.5

Table 20: Rheology of HVSE compared with Industry SE-1 and SE-2 at 150°F in 14.57 ppg polymer fluid with 100 ppb simulated drilled solids

Product	600 rpm (D.R.)	300 rpm (D.R.)	200 rpm (D.R.)	100 rpm (D.R.)	6 rpm (D.R.)	3 rpm (D.R.)
A	155.9	86.5	61.9	35.9	8.1	7.0
B	157.1	88.9	64.5	38.3	9.4	8.2
C	162.2	94.9	70.3	43.4	12.5	11.1
SE-1	279.8	178.3	140.1	97.2	44.3	41.6
SE-2	224.0	139.2	106.7	69.7	22.5	19.9