

Targeted Improvements in Chemistry of Common Fluid Additives Increase Temperature Stability of Non-Aqueous Drilling Fluids

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Abstract

Targeted optimization of common additives for non-aqueous drilling fluids, achieved through in-depth scientific research, has enhanced the composition of amidoamine-based emulsifiers. Combining these emulsifiers with optimized fluid loss additives and viscosity modifiers, improves performance, consistency, and extends the temperature performance window of invert emulsion-based drilling fluids. An improved method for emulsifier synthesis was developed and validated through an experimental approach under a wide variety of conditions. A similar approach was used to develop an optimized flat rheology fluid for subsea applications by simultaneous changes in multiple factors.

The newly developed emulsifier technology has been successfully scaled-up and produced in commercial quantities without any nonconformances. A new metric has been established to produce an emulsifier with the maximum active substances content without affecting physical properties, cost, and regulatory registration. Laboratory studies and initial field testing have demonstrated improvements in emulsifier shelf-life, product consistency, treatment levels reduction, and other benefits. The developed fluid exhibits a wide operational temperature window which can simplify logistics by eliminating displacement for specialized high-pressure/high-temperature (HP/HT) fluids that also improves the sustainability aspects of the drilling operation.

Introduction

Invert emulsions drilling fluids have dominated the high tier well construction market because of their superior performance, thermal stability, formation protection, lubricity, tolerance to contaminants, drilling performance, etc. Since the origin of invert emulsion fluids, advancements have mainly been made in changes of the base oil to improve the environmental profile, weighting materials, and the oil/water ratios. Not much has changed in the emulsifier chemistry used

in invert emulsions. The dominant emulsifier type used in invert fluids is an amidoamine based on diethylene triamine (DETA), tall oil fatty acids (TOFA), and maleic anhydride (Bistline et al., 1983 and Coates et al., 1988)). Recently, a series of articles (Khramov and Barmatov, 2021), (Khramov et al., 2020) (Khramov and Barmatov, 2021b) introduced how the design of experiments can be used for formulation of drilling fluids. As part of the effort, we compared different emulsifiers and demonstrated that an optimized emulsifier can be used at lower loadings to achieve a stable fluid versus a “conventional emulsifier”. Optimization of the amidoamine emulsifier was aimed at two goals, including improving stability of the system and optimizing product usage. In the study, presented in this paper, the final portion of the work related to emulsifier chemistry optimization and the effect of optimal emulsifier composition on temperature stability of non-aqueous i.e., invert emulsion fluid (NAF). The temperature stability of amidoamine-based NAF offshore fluids based on IO1618 are extended up to 365 °F. These emulsifier optimization efforts are bridging the gap between an amidoamine NAF and fit-for purpose exotic ultrahigh-pressure/ultrahigh-temperature UHPHT fluids.

Results and Discussion

Regardless of the long history of manufacturing of amidoamines, problems with consistency and quality still exist. One example of such an issue is separation during storage as shown in **Figure 1**. Frequently, separation issues are blamed on the weather (temperature) or insufficient quantities of pour point depressants blended in emulsifiers. The problem of separation during storage does not occur immediately after blending and formulating an emulsifier. The issue may well take place in a warehouse or on a rig, making it a nontrivial issue to assign a root cause for the problem.

While increased loading of pour point depressants can address the issue, it is a band-aid solution to a problem. Some

pour point depressants (PPD), such as low mass alcohols have low flash point, glycols such as butylenediglycol, butylenetriglycol, or other oxygenated solvents with acceptably high flash point can destabilize the emulsion which causes operational and environmental issues. An excess of PPD might not be an issue initially but as the emulsifier is depleted during drilling (Khramov and Barmatov, 2021b) and more emulsifier is added, PDD may well accumulate in the drilling fluid to undesirable levels.

The issue of emulsifier separation is not specific to one producer or one lot of a product. It is a systemic challenge that needs to be addressed. In addition to performance issues, separation is problematic when precipitated material plugs the bottom drain on the carryall, making the product difficult to be remove for use. While product performance might not be critical if the well that is drilled is not an HT or otherwise challenging well, inability to remove the product from a carryall due to plugging is an issue for all jobs.



Figure 1 – Separation of Amidoamine Emulsifier During Storage.

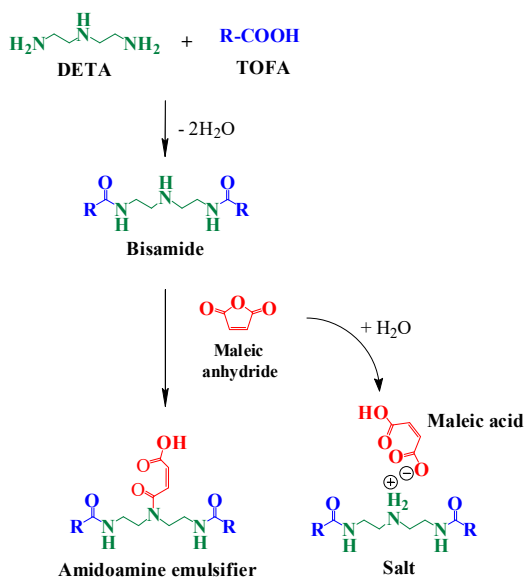


Figure 2 – Pathway to Synthesis of Amidoamines.

During previous efforts by the authors related to improvement of emulsifier quality, a new titration method (Khramov et al., 2020) was introduced that showed the amine value is consistently greater than reported by the previous

analysis methods. According to a generally accepted synthesis pathway (Figure 2) and understanding what makes an NAF amidoamine emulsifier, a high amine number is not expected for an amidoamine emulsifier. The final product should have a measurable acid number but zero amine number (Khramov and Barmatov, 2021). If an elevated amine numbers are found in the product it may well be an indication of an issue if the reaction proceeds according to Figure 2 or the final emulsifier molecule is different from the widely accepted structure.

Based on an amine number above greater than theoretical expectation, the authors proposed that precipitation occurs due to an incomplete reaction of the intermediate bis-amide with the maleic anhydride. A possible reason for separation is formation of a mixture comprised of amidoamine and an organic salt (Khramov et al., 2020) shown in Figure 2.

To comprehend the source of maleic acid that forms a salt that precipitates, a detailed review on the synthetic pathway of emulsifier production is required. Typically, maleic acid is not used for this process. More commonly, maleic anhydride is a raw material because it is more reactive than maleic acid. To appreciate how maleic acid forms in the reaction mixture, the authors focused on the first step of the synthesis, i.e. DETA and TOFA reactions. These reactions produce water that could hydrolyze maleic anhydride to form maleic acid. An amine intermediate is more reactive towards maleic anhydride than water which is the reason that the bulk reaction proceeds as intended; however, some maleic anhydride reacts with water and forms maleic acid.

Complete removal of water from the intermediate to prevent maleic anhydride hydrolysis is not feasible because removal of all water converts the bisamide to imidazoline (Bistline and Hampson, 1983). The reaction of imidazoline with maleic anhydride (Khramov and Barmatov, 2021) is extremely rapid, highly exothermic, and produces large volumes of CO_2 . While the imidazoline pathway can produce a functioning emulsifier the reaction of imidazoline with maleic anhydride is difficult to control on an industrial scale; therefore, it is undesirable. To prevent imidazoline formation, some water formed from TOFA-DETA condensation remains in the reaction mass so hydrolysis of maleic anhydride is unavoidable; thus a solution to an incomplete second step and resulting precipitation has to be addressed by other means. Efforts aimed at addressing the issue of incomplete reaction by means of reaction parameter optimization were completed in our

laboratories.

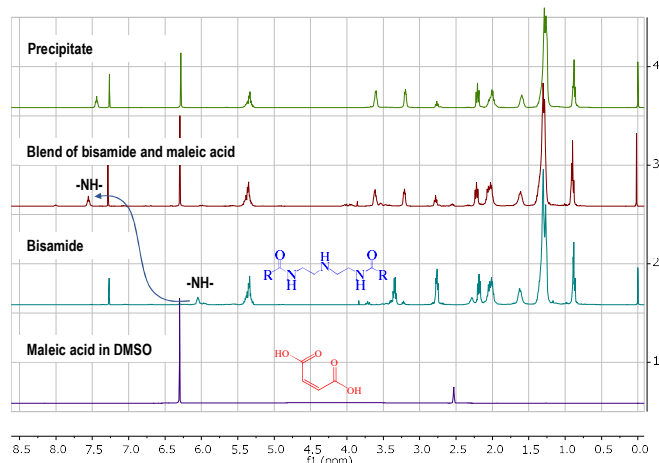


Figure 3 – ^1H NMR Spectra of Maleic Acid, Bisamide, a Blend of Bisamide and Maleic Acid, and Isolated Precipitate.

Precipitate analysis of precipitate shown in **Figure 1** obtained by NMR spectroscopy confirmed that this material corresponds to salt of bisamide and maleic acid as shown in **Figure 3** and **Figure 4**. An identical ^1H NMR and heteronuclear multiple-bond correlation (HMBC) spectra was obtained by combining bisamide intermediate with maleic acid in 1:1 molar ratio confirming correct structural assignment for the material isolated from commercial separated emulsifier samples. HMBC also shows no cross-peaks between bisamide and maleic acid substructures, which suggests that the precipitate is a blend (not chemical compound) of starting materials.

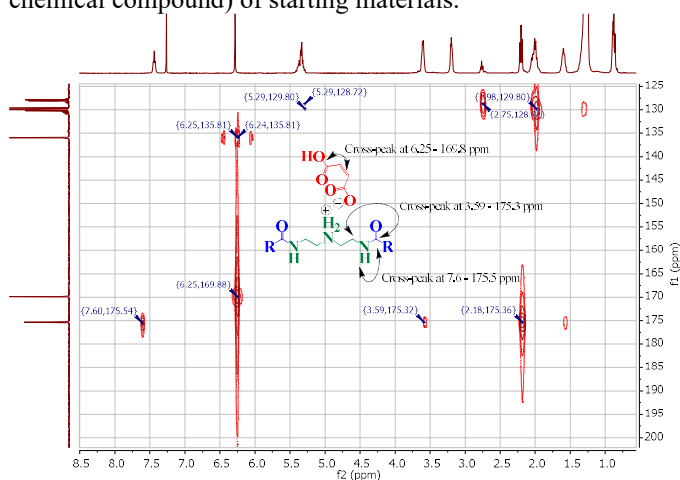


Figure 4 – HMBC Spectroscopy and Structural Assignment of Precipitate as Salt of Bisamide and Maleic Acid.

Gas chromatography-mass spectrometry (GC-MS) confirmed presence of maleic acid. This investigation confirmed the issue of incomplete reaction between maleic anhydride and a bisamide intermediate. The authors initiated a development program to resolve the issue of emulsifier separation during storage. During our investigation (Khramov and Barmatov, 2021) we determined that the reaction and the

final emulsifier structure differs from what is typically reported in the literature (Coates et al., 1988). In addition to resolving the issue of precipitation, we optimized the yield of active emulsifier ingredient. Based on these achievements, we were able to extend the performance envelope of a NAF formulated around newly optimized chemistry.

After concluding our optimization studies (**Figure 5** and **Figure 6**), the authors confirmed by previously described analytical methods that we successfully completed the synthesis reaction and there was no remaining salt left that caused precipitation. Furthermore, we identified a key chemical structure responsible for supporting an emulsion (Khramov and Barmatov, 2021). This structure is different from the product shown in **Figure 2**. Based on this finding we prepared to evaluate the benefit of improved emulsifier based on performance in NAF.

A series of evaluations of various iterations of emulsifier chemistry were completed by formulating a NAF in IO1618 and testing it at 325 °F. Rheology and fluid loss properties after 16-hour hot roll (AHR) were recorded. It was observed that rheology parameters were similar for the entire effort related to emulsifier chemistry optimization but fluid loss testing showed differentiation between experimental emulsifiers. **Figure 5** shows a trough where fluid loss is minimal. Under- and over-reaction show degradation in HPHT performance.

Various advanced techniques were used to follow the reaction pathway and elucidate the mechanism and resulting structures (Khramov and Barmatov, 2021). For ease of manufacturing, quality assurance (QA) was based on simple methods, such as acid number, amine number, and viscosity. Correlating the results in **Figure 5** and **Figure 6** we determined the preferred properties of the emulsifier and what manufacturing conditions to follow to achieve the desired outcome.

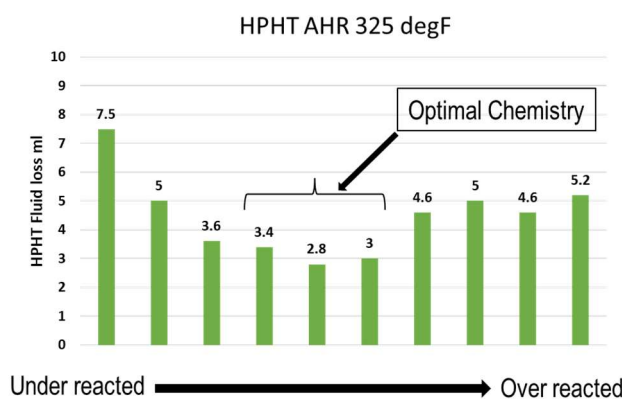


Figure 5 – Fluid Loss Results of Emulsifiers Based on the Optimization Effort. 325°F HP/HT test.

The optimized chemistry attempt eliminated the issue of emulsifier separation during storage. The authors believe addressing the problem by means of the improved amidoamine synthesis pathway is the preferred solution over other methods such as increase in PPD addition. Because PPD can have a

destabilizing effect on a NAF emulsion, double benefits are obtained from our attempt at better chemistry and reduction in pour point additive.

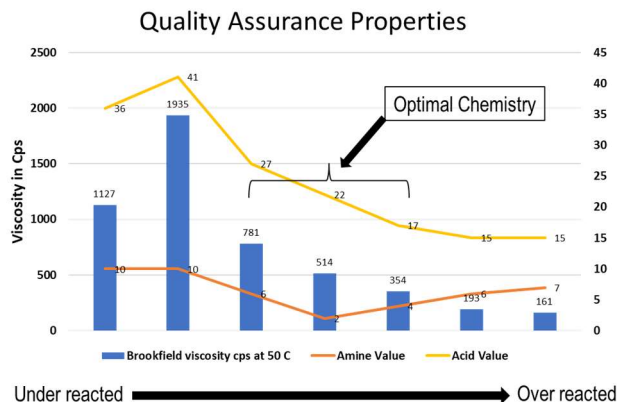


Figure 6 – Quality Assurance Properties Developed During Emulsifier Optimization.

In addition to resolving the issues related to emulsifier storage and formulation, it is expected that issues related to performance consistency are also eliminated. The authors have previously mentioned that the intermediate from incomplete reaction of amidoamine can function as an emulsifier when combined with a fatty acid in 1:1 molar ratio to form a salt (Khramov et al., 2020). However, these amine salts are not stable and in the presence of lime they break down to neutral bisamide and a calcium soap of fatty acid. Formation of bisamide salt with a fatty acid might be one of the reasons why the addition of wetting agents improves emulsion stability; however, this improvement is only temporary in nature and not temperature-tolerant.

In present efforts related to reducing precipitation during storage, we optimized the reaction between bisamide and maleic anhydride and acid and reduced the amine number to a low value (see **Figure 6**). This low amine number should improve consistency of emulsifier performance. Completing the reaction between the intermediates also increases the amount of active emulsifier available and should allow for reduced treatment levels during drilling (Khramov and Barmatov, 2021). Finally, with improved chemistry the authors believe that improved performance can be achieved based on indications in previously published work (Khramov et al., 2020), (Khramov et al., 2020), and (Khramov and Barmatov, 2021b) such as reduction in emulsifier loading and increase in maximum temperature at which the NAF will perform.

With an optimized emulsifier, we completed a design of experiments (DOE) based performance evaluation similar to previous efforts (Khramov et al., 2020). A key difference from previous work where only components were changed in a formulation, in this current study we included hot roll and filtration temperatures as variables. The advantage of using DOE for this study was the ability to evaluate multiple factors at the same time and to improve our confidence in results because conclusions are based on simultaneous analysis of 50+

mud samples. Optimization of offshore NAF was conducted using IO1618 as the base fluid. The goal of the effort was to evaluate temperature limitation while maintaining flat rheology and low equivalent circulating density (ECD) of the fluid. Other regions, not needing IO1618, or not requiring flat rheology fluids may be able to further extend temperature performance of their formulations.

Table 1 – Formulation Details.*

Amodril 1000	146
Emulsifier	8-14
Wetting agent	0-3
Rheology control additive	3.00
Lime	2-10
25% CaCl ₂ Brine	68.0
Synthetic fluid loss additives	2-6
Organophilic tannin	0-10
Suspension additive	6-14
Rheology modifier	0.5-3
Micronized barite	349
Hot roll and test temperature °F	330-380
SWR, %	80.0
Mud weight lbm/gal	14.00

*Quantities are lbm/bbl or g/350ml

A general formulation used for DOE optimization is shown in **Table 1**. The focus in this effort was on 14 lbm/gal fluid at 80/20 oil-water ratio (OWR). We have previously successfully included fluid density and OWR as factors in the DOE, but to limit the amount of work for this demonstration effort we focused on a single fluid weight and single OWR.

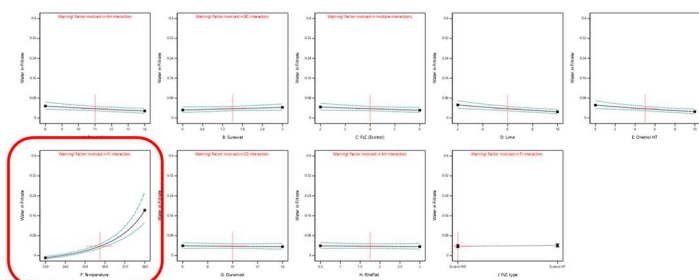
Products that were used as variables have ranges of concentration included (min/max) instead of a fixed value. We adjusted the amount of base oil and weighting agent to maintain fixed values of density and OWR.

The present study was setup to evaluate two-factor interactions and required a minimum of 47 runs. A total of 56 runs were completed to generate a reliable model, include replicates, and to improve signal-to-noise ratio. Additional details on design of experiments are found in our previous publications (Khramov et al., 2020) and (Khramov et al., 2020).

Because the main criteria on this study were to comprehend temperature stability, we evaluated the design with focus on HP/HT filtrate and the appearance of water in the filtrate. We ran the fluid loss test on a WFAO-A disk (5 µm) for 60 min with 500-psi pressure differential. After creating the DOE models, we determined that temperature is the single most important factor on the appearance of water in the filtrate as shown in **Table 2**. The quantity of emulsifier or wetting agent did not have a significant bearing on these results because we have previously shown that it takes very little emulsifier to stabilize an emulsion. 4- to 5-lbm/bbl emulsifier would be sufficient to stabilize a typical NAF emulsion, and, in the present study, we used 8- to 14-lbm/bbl emulsifier which is in

excess of minimum amount of emulsifier that was found to be absolutely necessary (Khramov and Barmatov, 2021b).

Table 2 – DOE Model Analysis (Including 95% Confidence Intervals) for Water in Filtrate after 60-min HP/HT Test.



A binary logistic regression (Figure 7) shows that likelihood of water above 370°F. Adjusting factors used in the model it is possible to shift the curve further to the right but in all cases, water in the filtrate at >375°F was expected according to our analysis.

Based on our analysis of DOE data we determined that a stable fluid could be formulated at 365°F with good rheology profile and no water in the filtrate when HP/HT was performed on a ceramic disk. Because testing is done based on laboratory fluids, we selected 365°F as the recommended maximum temperature for the fluid. This temperature is sufficient to cover the vast majority of IO1618-based NAF in the Gulf of Mexico.

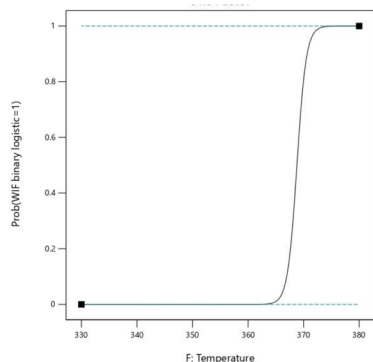


Figure 7 – Binary Logistic Analysis of Water in the Filtrate with Temperature as a Factor. The Y-axis is 0 (0% Chance of Water in the Filtrate) to 1 (100%).

A confirmation of the DOE model analysis was performed by numerically optimizing the system for lowest viscosity and a flat rheology profile with temperature. Formulation was proposed using a computer-based model the authors created using constraints such as flat rheology profile, no water in the

filtrate, and acceptable rheology values (see Table 3).

Table 3 – Fluid Properties Predicted Based on a Numerical Model. Point Prediction

Two-sided Confidence = 95% Population = 99%

Analysis	Predicted Mean	Predicted Median*	Observed	Std Dev	SE Mean	95% CI low for Mean	95% CI high for Mean	95% TI low for 99% Pop	95% TI high for 99% Pop
R600@40F	223.042	222.46		16.145	N/A	203.17	244.858	163.013	303.587
R5@40F	6.54175	6.43708		1.18452	N/A	4.87905	8.77108	2.77446	14.9348
10min@40F	13.1618	12.9378		2.45945	N/A	10.4762	16.5358	5.8281	28.7208
Untitled	93.5994	93.5994		15.1028	9.80432	73.6523	113.546	28.0846	159.114
6@150F	8.48573	8.06062		2.79202	N/A	5.78791	12.441	2.08355	31.1842
10min@150F	19.6472	17.7661		9.27724	N/A	11.564	33.3804	2.68736	117.452
HPHT 1hr	4.1672	4.03947		1.0562	N/A	3.26973	5.311	1.46651	11.1266
Water in Filtrate*	0.0274643	0.0246435		0.0172236	N/A	0.0148667	0.041771	-0.0136528	0.182219

The actual results matched the DOE-predicted results well. Formulation details and results are shown in **Error! Not a valid bookmark self-reference.** which demonstrates use of DOE models to optimize complex systems based on a large number of variables. In the results we see that there is no water in the filtrate after the HP/HT test which was an important design criterion. Furthermore, static aging of the fluid in an aging cell showed low static shear values meaning the fluid does not gel up during high temperature aging and furthermore, rheology of the fluid after five-day and seven-day static test was comparable to AHR rheology also indicating good fluid stability, and the Emulsion Stability (ES) after seven days remained high at 774 volts.

Table 4 – Formulation Details and AHR Rheology results.

Amodril 1000	139
Emulsifier	10.00
Wetting agent	0.00
Rheology control additive	3.00
Lime	10.00
25% CaCl ₂ Brine	64.5
FLC	4.00
Organophilic tannin	10.00
Suspension additive	14.00
Rheology modifier	1.00
Micronized barite	333
SWR, %	80.0
Mud weight, lbm/gal	14.00

	AHR		5-day		7-day	
Heat Aging Temp, °F	365		365		365	
Heat Aging, hr	16		120		168	
Static/Rolling	D		S		S	
Rheology Temp, °F	40	150	40	150	40	150
R600, °VG	237	74	261	76	273	78
R300, °VG	130	44	143	45	150	46
R200, °VG	91	33	100	33	104	34
R100, °VG	49	20	54	20	57	20
R6, °VG	7	7	6	6	8	6
R3, °VG	5	7	5	5	6	6
PV, cP	107	30	118	31	123	32

YP, lbm/100ft ²	23	14	25	14	27	13
LSYP, lbm/100ft ²	3	7	3	5	4	5
10-sec Gel, lb/100ft ²	7	10	7	10	8	11
10-min Gel, lbm/100ft ²	12	38	22	35	22	39
Static Shear, lbm/100ft ²				<10		<10
ES @150°F, V						774
HTHP Temp, °F	365	365				
HTHP Time on disk	30min	60min				
HTHP FL, ml	3.3	4.4				
Water in HTHP, ml	0	0				

If HP/HT filtration is performed on paper, as it is frequently carried out in the laboratory, maximum temperature at which we observe no water in the filtrate is ~380°F (see **Table 5**).

Table 5 – Formulation and Performance Data for a NAF Targeting 380°F Temperature.

Amodril 1000	139
Emulsifier	8.20
Wetting agent	1.26
Rheology control additive	3.00
Lime	10.00
25% CaCl ₂ Brine	64.4
FLC	6.00
Organophilic tannin	10.00
Suspension additive	10.00
Rheology modifier	0.50
Micronized barite	336
SWR, %	80.0
Mud weight, lbm/bbl	14.00

Heat Aging Temp, °F	380		
Heat Aging, hr	16		
Static/Rolling	D		
Rheology Temp, °F	40	100	150
R600, °VG	246.8		122
R300, °VG	137.1		75.6
R200, °VG	97.5		58.1
R100, °VG	55.2		36.5
R6, °VG	8.4		10.9
R3, °VG	6.2		9.3
PV, cP	110	0	46
YP, lbm/100ft ²	27	0	29
LSYP, lbm/100ft ²	4	0	8
10-sec Gel, lbm/100ft ²	10		9.4
10-min Gel, lbm/100ft ²	16.4		13.5
HTHP temp, °F	380	380	
HPHT time	30min	60min	
HTHP FL, ml (disk)	3.7	4.6	
Water in filtrate, ml (disk)	trace	0.07	
HTHP FL, ml (paper)	1.5	2	
Water in HTHP filtrate, ml (paper)	0	0	

To demonstrate additional potential of the fluid, same formulation was prepared and tested at 401°F, which under extrapolation of DOE data would show some water in the filtrate. As expected, there were traces of water in filtrate (see **Table 6**). Based on results shown in **Table 6** we see that rheology AHR is reasonable and within the DOE model predictions even if this fluid is built on an extrapolation of temperature beyond the model design. As expected, water in the filtrate was observed, indicating some instability of emulsion; however, the volume of water is not large. When an HP/HT test is conducted on paper for a typical 30-min test, quantity of water in the filtrate is further reduced. These results open the possibilities for further optimization of the system to extend the temperature limitation beyond the 365- to 370°F range limit that we currently established. For regions where NAF fluid is not required to use IO1618 or have a flat rheology profile, it may well be possible to formulate a stable fluid for bottom hole temperature exceeding 365°F. However, even a 365°F limitation that we estimate from our model analysis is sufficient to bridge the gap between the common amidoamine-based NAF used for Gulf of Mexico and ultrahigh temperature NAF that are also commercially available.

Table 6 – 401 °F Testing Results.

Heat Aging Temp, °F	401		
Heat Aging, hr	16		
Static/Rolling	D		
Rheology Temp, °F	40	100	150
R600, °VG	259.3		134
R300, °VG	147.2		81.3
R200, °VG	105		60
R100, °VG	58.5		37.9
R6, °VG	10.2		12.7
R3, °VG	7.6		11.1
PV, cP	112	0	52
YP, lbm/100ft ²	35	0	29
LSYP, lbm/100ft ²	5	0	10
10-sec Gel, lbm/100ft ²	12.3		12.3
10-min Gel, lbm/100ft ²	20.2		14.3
HTHP temp, °F	401	401	
HTHP time	30min	60min	
HTHP FL, ml (disk)	4	5.1	
Water in filtrate, ml (disk)	0.2	0.3	
HTHP FL, ml (paper)	2	2.7	
Water in filtrate, ml (paper)	0.1	0.15	

A discussion on fluid loss as a criterion for stability is not complete without considering fluid loss additives. We selected appearance of water in the filtrate as a stability factor. However, this assumption is meaningless without addressing total filtration volume. Stable emulsions function as filtration aids themselves but if total HP/HT volume is larger than typical limit of 4ml in 30-min, this means that the oil phase is removed

with the filtrate and the OWR ratio of the remaining fluid changes to less oil and more water. At some point, if filtration losses are too large, the OWR in the filter cake will be low and water droplets will coalesce (Kocherginsky et al., 2003).

Analysis of the DOE model for water in the filtrate and reduction in total fluid loss showed that at 380°F an organophilic tannin works better than synthetic fluid loss polymers (see **Figure 8**) for reduction of HP/HT filtrate volume. Additives such as styrene-acrylate copolymers are less effective than organophilic tannins at reducing fluid loss. It is possible that these materials degrade at extreme temperatures while organophilic tannin remains stable. Additionally, when the HP/HT test was extended past the typical 30-min duration we observed that synthetic additives merely reduce the rate of HPHT filtrate appearance but never stop the flow completely while organophilic tannin is able to shut off filtration permanently. Tannin-based fluid loss control (FLC) is a beneficial additive to be able to formulate an amidoamine-based NAF for high-temperature applications.

Finally, with an optimized emulsifier and proper selection of fluid loss additive, we realized a series of benefits for HT fluid such as stability of the system across a wide range of concentrations of many additives. We did not see issues related to increasing lime content which means that hydrolysis of the emulsifier is not a major factor in emulsion destabilization. Our data also shows that 8 lbm/bbl of emulsifier is sufficient to stabilize the formulated system even at high temperatures. We have previously discussed the effect of emulsifier on NAF stability and concluded that 5- to 6 lbm/bbl of amidoamine is sufficient to stabilize the fluid if the emulsifier does not degrade. Our current data supports this conclusion; i.e., in the range of 8- to 16 lbm/bbl emulsifier, 8 lbm/bbl is sufficient due to the exceptional stability of the emulsifier we developed.

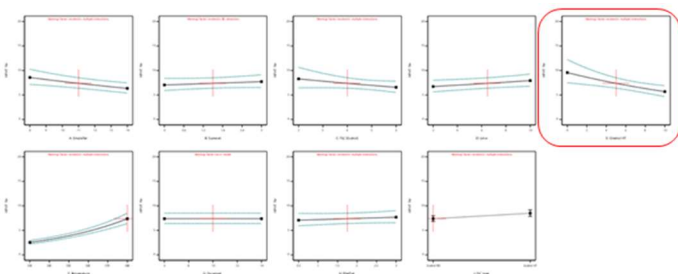


Figure 8 – HP/HT volume DOE Analysis (All Factors). Organophilic Tannin Additive is Highlighted.

Conclusions

We completed our extensive investigation related to emulsifier separation during storage and determined that the problem is related to incomplete conversion of the intermediate to target product. During this investigation we also determined that the reaction pathway to synthesize amidoamine emulsifier is more complex than previously considered and composition of actual emulsifier chemistry is poorly understood.

We optimized the manufacturing parameters of the emulsifier to stop the separation process and, simultaneously,

to improve the emulsifier consistency and quality. We evaluated the performance of a new, modified, emulsifier across the broad range of formulations using DOE and demonstrated that with the improved amidoamine emulsifier we can formulate flat rheology profile NAF up to 365°F. Our new optimized emulsifier is highly effective, and performance is relatively insensitive to changes in the emulsifier concentration across a wide range or changes in concentrations of other components. This result provides the formulator a large option window to optimize a fluid for their specific requirements without running into issues.

Our efforts aimed to further extend the performance envelope to 401°F showed some instability in emulsion but also a potential to extend the temperature performance window by incorporating a high performing fluid loss control agent such as organophilic tannin.

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