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Evaluation of Laboratory Performance Tests for Drilling Fluids

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Abstract

Testing of drilling fluids in the laboratory is an early and essential part in developing a new drilling fluid. There are different types of tests that could be split into two general categories: screening tests and advanced tests. A series of basic screening tests are performed because these tests are simple to execute, inexpensive, and they give some idea about fluid performance. More advanced testing such as pore pressure transmission, thick wall cylinder tests, and many others are available and can provide better information about fluid performance, but these tests are too low throughput to be used as screening tools for evaluation of new drilling fluid additives. Objective of this paper is to discuss the resolution of common lab tests to improve our ability to interpret the results. Experimental design and statistical data treatment were applied to standard lab testing protocols to highlight what fluid components affect results the most and what are typical variabilities of each test method and what are confidence intervals for the trends that we established. Bulk hardness, shale hydration, shale dispersion, linear swell meter, accretion, and bentonite inhibition were studied. All lab tests have some inherent variability due the test itself and due to the variable nature of shales and clays that are tested so data interpretation should be done with variability in mind. Details presented in this paper can help practitioners place increased emphasis on a specific test depending on what their goal is and de-emphasize distracting and irrelevant test data. Improved understanding of the error bars on lab tests will make comparisons between different products more meaningful which will aid in developing of higher performing aqueous fluid systems.

Introduction

What is a laboratory test that will provide an answer that correlates to the field performance is a challenging question to answer. A series of papers discussing the value of lab test have been previously published by van Oort et al 2003, 2016, 2018. In summary of previous literature publications, it can be stated that importance of representative rock samples is paramount and the preferred rock-fluid interaction tests are pore pressure transmission (PPT) and thick wall cylinder test (TWC). However, the proposed test methods and materials are more suitable for the final validation of the system to be taken for the field. Due to complexity, cost, duration of the test and availability of the representative rock samples, neither TPC or TWC can be used as a screening test to select preferred products from a myriad of commercial or experimental products. We believe that the legacy methods used for laboratory fluid testing are suitable as screening tools and many in the industry still rely on the data provided by those tests. Until the legacy test methods disappear entirely, we would like to provide in depth analysis on what do these methods can show us. In this paper we focus on laboratory testing of shale-fluid interaction for water-based drilling fluid but the concepts and methods we share are applicable to nonaqueous fluids (NAF) as well.

Problem Statement

6-speed rheology, shale dispersion test, shale accretion test, bulk hardness test, linear swell meter test, API and HPHT fluid loss, and bentonite inhibition tests are common test methods used in the lab for decision making. The reproducibility for each test is not always discussed when interpreting the test data. Also, with limited number of data points, it becomes difficult to decouple chemistry versus viscosity effects or correctly attribute performance to a certain component.

In this paper we will focus on discussion of the variability of common tests due to shales variability and test equipment. Also, it can be stated that the value from laboratory experiments is to establish trends in performance rather than look at absolute values. In this study we rely on experimental design as a method of establishing more reliable trends, look at synergies between components, and improve our confidence when assigning performance benefits to various additives. The conclusions in this paper will help the industry differentiate better between the output and the noise of these measurements. We will also discuss the results of experimental design approach and demonstrate how fluid additives affect testing properties such as shale hydration, dispersion, bulk hardness, and rheology based on the bentonite inhibition test, or linear swelling.

Discussion and Results

We apply design of experiments (DOE) methodology in this study. This approach has worked well for us when developing new products and optimizing NAF performance [Khramov et al, 2020, 2021]. Changing multiple parameters at the same time and creating a comprehensive drilling fluid digital avatar is a preferred approach when dealing with a complex product like a drilling fluid with multiple components and many output variables. There are vendors with commercially available software packages that can facilitate design of the test matrix. In our previous papers we also described in detail our approach to setting up DOE studies [Khramov et al, 2020, 2021] so we will not include detailed description of step-by-step design of the study. We will describe the results and include relevant statistical model analysis when necessary.

Rheology

Typical rheological measurements using 6-speed coaxial couette viscometer are highly repeatable and reliable, if temperature of the sample is controlled well. This example of model creation and data analysis is presented to m mainly illustrate how design of experiments approach is utilized for our studies. In this study, we chose many numeric and categoric variables to create the study matrix (Figure 1). Total number of experiments used to create the model was 67 individual experiments

Variable factor	Minimum level	Maximum level
Polymeric sealant	0	20 ppb
Fluid Loss Additive	0	10
Asphalt	0	15 ppb
NaCl	0	20 %
Wellbore Stabilizer	0	15
Viscosifier	0.3	1.5
API evaluation clay	0	30
Fluid density	10	14
Fluid Loss Additive	Product A, B, C	

Figure 1. Design factors (will sensor names for the actual paper).

After 67 fluids were mixed and their properties were measured, a set of models were created based on input variables (Figure 1) and output variables which were 6-speed viscosity measurements. Rheology measurements showed good statistical model analysis for R600, and R6. Model fit was acceptable based on fit statistics (Figure 2).

	R^2	Adjusted R^2	Predicted R^2
R600	0.9731	0.9605	0.9394
R6	0.9613	0.9420	0.9106

Figure 2. R600, R6, model fit statistics respectively.

As expected, the response of the model for R600 was dominated by Biopolymer and, to a lesser extent, starches used for fluid loss control (Figure 3).

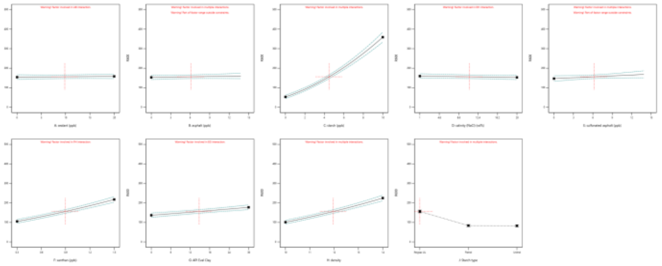


Figure 3. Model for R600 at 120℉ AHR.

Similar approach to experimental setup, model fitting, and analysis was done for all other lab tests we evaluated in this paper.

Linear Swell Meter

Moving on to shale-fluid interaction measurement, we evaluated the linear swell meter test using design of experiments. For linear swell meter testing, shale is ground to fine powder and then compressed into a pellet. The pellet is placed into a hollow steel cylinder and change in shale volume is recorded by measuring linear displacement of a plunger placed on top of the clay pellet. In this study we used Oxford shale. Oxford shale contains approximately 23% smectite and, therefore, has moderate swelling tendencies. In our study of the swelling range from best to worst experiment was 5-35% swelling i.e., the experimental setup had good resolution to reach a conclusion on the linear swell test. The experimental test matrix is shown in Figure 4. Test conditions were conducted at 150°F, 200 PSI pressure, and all fluids were at pH 9.6. Swelling test was conducted for 16 hours which was sufficient to reach steady value of linear swelling. The experiment was setup with a constraint that the salt can be NaCl or KCl or a mixture up to 20 wt. % total salt content. The study consisted of 40 separate experiments so we can be confident that we are generating reliable trends.

Variable factor	Minimum level	Maximum level
NaCl	0	20 %
KCl	0	20 %
Shale Inhibitor	0 ppb	10 ppb
Viscosifier	0 ppb	1.5 ppb
FLA	0 ppb	3 ppb
Non-aqueous additive	0 ppb	10.5 ppb
Polymeric sealant	0 ppb	15 ppb

Figure 4. Linear swell meter test matrix.

Our model fit statistics show that the model we selected describes the relationship between the variables and the results well for the final 16-hour swell and less so for 1-hour swell.

	R^2	Adjusted R^2	Predicted R^2
1 hour swell	0.7699	0.6877	0.5446
Final swell	0.9103	0.8863	0.8250

(16 hrs.)			

Figure 5. Linear Swell Meter Fit statistics.

1 hour swell Predicted R-squared was only 0.54 which is lower than model fit for final swell model. The difference in model quality can be explained by presence of variations in setting up the test and those variations being a significant portion of the test time if we are looking at 1 hour swell test. Conducting a swelling test until clay pellet stops swelling is a common approach in the industry. In this example, we are comparing models for 1 hour and 16 hour swelling results to demonstrate how trends, and possibly conclusions, may be different due to variability related how test is conducted. For analysis of the results, we focus on the final (16 hour) swell model that is shown in Figure 7. The trends are in black lines and 95% confidence intervals is in teal dashed lines. The narrowness of confidence bands shows a reasonably reliable model. Another way to demonstrate reproducibility of linear swell measurement is to look at replicates from the DOE study (Figure 6). The variability is approximately 1% in the test. In our case this error has moderate effect on the result since the range of swelling for Oxford shale in our study 5-35% but if the swelling is small 0-5% when comparing 2 fluids then error of 1% is significant and conclusions should not be made based on a single measurement.

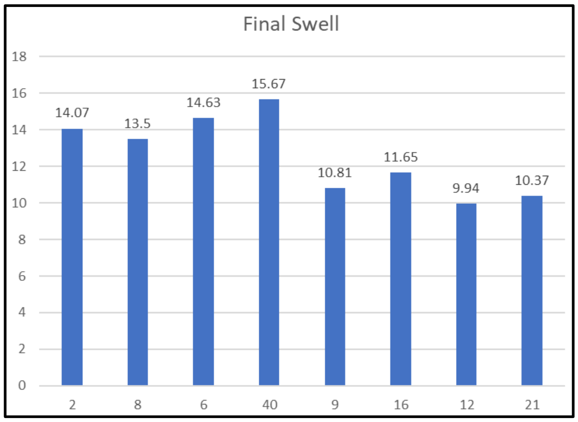


Figure 6. Pairwise comparison of replicates 2 and 8, 6 and 40, 9 and 16, 12 and 21.

From the model analysis (Figure 7, Figure 8) we see that 95% confidence intervals are narrow around salt and the slope for salt is largest. This suggests that linear swell meter study mainly responds to salt concentration and not very sensitive towards other components of the fluid.

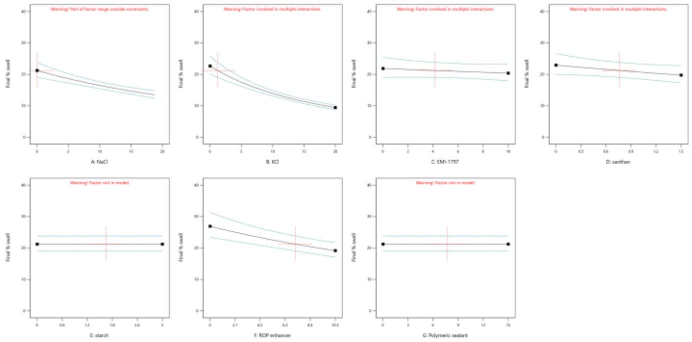


Figure 7. Linear swell meter – factors affecting linear swelling.

Worth noting that in linear swell meter test as shown in the Figure 7 amine inhibitor has almost no effect on swelling in the range of 0-10 ppb in the fluid is somewhat viscosified (0.5 ppb xanthan). In an un-viscosified fluid indicated by red crosshair at zero for viscosifier there is some limited effect of amine inhibitor (Figure 8).

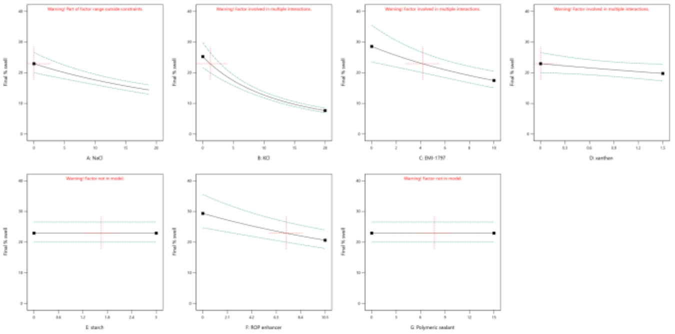


Figure 8. Amine inhibitor effect on linear swelling in ultra-low viscosity fluid (brine).

Since typical drilling fluids will contain viscosifiers, we can say that in this linear swell meter study with many factors only salt matters on degree of swelling. This conclusion is not universal for all shales, but it is indicative that to reach proper conclusions and separate signal from noise to reach reliable conclusions is preferred. Also, this does not mean other additives besides salt are ineffective at preventing swelling; however, it does suggest that using linear swell meter test may not be the right test to evaluate effects of fluid components other than salinity.

Another set of observations that can be made because we created a comprehensive response surface is looking at effect of NaCl vs KCl in the presence or absence of amine. In our model for linear swell, observe that KCl is more effective at reduction of swelling than NaCl and this effect remains whether we use amine inhibitor or not. The observation is interesting, considering that a common explanation why KCl works well is potassium cation exchanges with Na ions at available sites in the clay. This process of cation exchange for potassium reduces clay swelling [O'Brien 1973, Uti et al 2013]. With amines having stronger affinity for active cation-exchange sides [Cui et al 2010, Fletcher et al 1989], suggests that with amine present, it should be irrelevant whether Na or K salts are used because cation-exchange sites are saturated with amines and, therefore Na vs K effects should not be as apparent and the swelling of clay with NaCl vs KCl is expected to be similar when cation exchange sites are taken up by amines; however, that is not the case according to our study. This means that another mechanism is at least partially responsible for clay swelling.

Shale Hydration

Shale hydration study is typically performed by hot rolling shale cuttings in drilling fluid, recovering cuttings, and drying them to measure moisture content. To understand which components affect shale hydration, we completed an extensive study that included numerical and categorical factors - shale inhibitor, synthetic polymer viscosifier, and brine type (Figure 9). Other typical components of the drilling fluid were also present – xanthan, starch, barite, ROP enhancer, and lubricant. Since these components remained fixed for this study, their effect is not discussed in this evaluation. This study included 45 experiments to create a trend.

Variable factor	Minimum level	Maximum level
Shale inhibitor	0.25 vol. %	4 vol. %
Synthetic Polymer Viscosifier	0.25 ppb	4 ppb
Inhibitor type	A, B, C, or D	
Brine type	1 % NaCl or 7% KCl or 20 % NaCl	
Shale type	Arne	Oxford

Figure 9. Study parameters for the shale hydration evaluation

The model fit was not great relative to our models on other properties. Fit statistic for the best model we created is shown in Figure 10.

	R^2	Adjusted R^2	Predicted R^2
Shale Hydration	0.6481	0.5576	0.4260

Figure 10. Fit statistic for shale hydration model

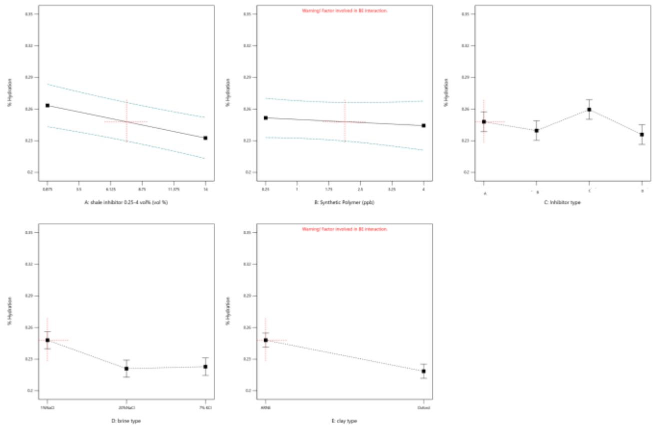


Figure 11. Model on parameters that influence shale hydration.

In our model (Figure 11) we see broad confidence intervals. Some effect due to quantity of shale inhibitor but not the type of inhibitor i.e., we cannot differentiate confidently which inhibitor is best. The main parameter affecting shale hydration in this relatively narrow range is salinity of the fluid. Since the hydration is measured by water mass loss, a calculation for hydration with different salinity brines suggests that volumetrically we have similar amount of brine ingress into shale pores. Higher salinity brines simply have less free water to lose. Challenges with analyzing hydration results and effect on hydration from something other than salts can be explained by looking at Figure 12. This is a plot of predicted versus actual results and different color corresponds to two shale types we used. Looking at x-axis we see that Oxford hydration clustered around 20-22% moisture regardless of variables in formulation. Arne showed slightly better differentiation but even then, the range was only 20-27% after excluding a few obvious outliers. The data shows based on clustering that Arne and Oxford are different from each other but for each clay type hydration does not change significantly regardless how we vary other components in the study. Model in Figure 11 gives an appearance that shale inhibitor reduces hydration but worth noting that the range of inhibitor loading was 0.88 to 14ppb in other words a very wide range of inhibitor loadings only to achieve a reduction of Arne hydration of 3% between best and worst result. For comparison, starting hydration of Arne shale used for this test was about 12-14% which means amine does not help reduce hydration much relative to the initial state. The effect, or lack thereof, from amine is not entirely surprising since we used Oxford and Arne shale and their CEC is low which means that not much amine is needed to inhibit these cuttings.

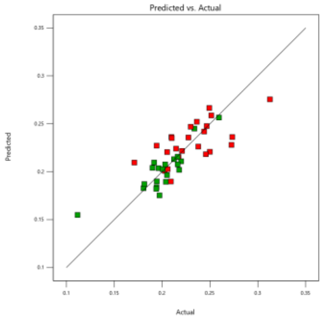


Figure 12. Predicted versus measured for shale hydration (green is Oxford and red is Arne shale).

Shale Dispersion

Typical shale dispersion test is done by adding 30 or 40 grams of 4-6 mm cuttings to 1 lab barrel of formulated drilling fluid and hot rolling the sample for 16 hours after which cuttings are removed by filtration on 2mm sieve. Then washed to remove excess mud and rolled on a paper towel to remove residual mud and brine form surface. After normalization for moisture content before/after hot roll percentage of cuttings recovered is recorded. Typically, recovery of shales is high in most cases when fluid is hot rolled at 150 or 180°F. In a DOE study we included a range of factors which resulted in a matrix of 30 experiments (Figure 13). One important factor is to attempt to decouple effect of chemistry versus viscosity by including xanthan viscosifier and a polymeric viscosifier. Model fit statistics is shown in (Figure 14).

Product	Low Range	High Range
Xanthan	0.2 ppb	1 ppb
Inhibitor	0.5 ppb	10.5 ppb
Mud density	9 ppg	12 ppg
Synthetic polymer	0 ppb	2 ppb
API Eval. Clay	0 ppb	35 ppb

Figure 13. Variables in a dispersion study

	R^2	Adjusted R^2	Predicted R^2
Shale Recovery	0.8764	0.8370	0.7331

Figure 14. Shale recovery model analysis.

Analysis of the results (Figure 15) shows that xanthan and synthetic polymer have similar effects as does mud weight. This suggests that at least by this testing protocol, reducing mechanical forces on the cuttings displays the dominating effect on the recovery of shale. Also, after decoupling viscosity from chemistry effects for biopolymer vs synthetic polymer we conclude that viscosity effect dominates and there is not a strong chemistry interaction between polymer and shale. Only under a very narrow range of highly specific scenarios, we were able to establish that synthetic polymer can contribute to increased shale recovery when inhibitor was not present.

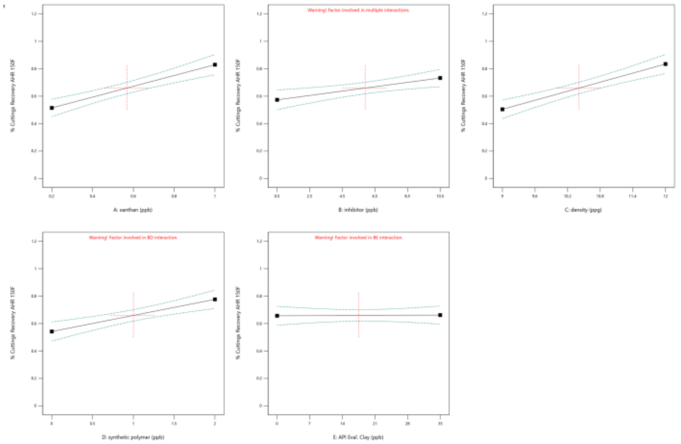


Figure 15. Model for shale dispersion testing.

The study of shale dispersion tests suggests that the test is not the most suitable method to evaluate additives designed to prevent shale dispersion tests unless a lot of care is taken to decouple chemistry from viscosity effects.

Shale Accretion

Shale accretion test is performed by adding cuttings to 350ml fluid in the lab and placing a steel tube in a jar and rolling the sample for specified amount of time which is commonly measured in minutes and after which the tube is removed and amount of stuck cuttings is measured. The variability of the test parameters can have significant influence on the results to the point, the test is frequently called a qualitative test. We completed a study to understand if quantification of accretion is possible and what parameters affect the quantitative output of the test. The variables selected for the study is shown in Figure 16. Arne and Oxford shale was used for the study. The model consisted of 24 separate experiments.

Product	Low Range	High Range
Anti-accretion additive	0 ppb	20 ppb
Test length	5 min	15 min
Inhibitor amount	0 ppb	10 ppb
Xanthan	0 ppb	1 ppb
pH	8.5	11

Figure 16. Variables in accretion test study.

Model fit statistics is shown in Figure 17.

	R^2	Adjusted R^2	Predicted R^2
Arne accretion	0.9678	0.9178	0.7552
Oxford accretion	0.7208	0.6742	0.5644

Figure 17. Fits statistics for the model created to study accretion test.

Results for Oxford shale accretion tests are shown in Figure 18. Time is the main factor affecting accretion with small contributions from viscosifier. Increased viscosifier possibly making the shale stickier. Time factor suggests that at the scale of this test Oxford shale increases hydration and comes softer so as test runs longer, accretion is higher. Also, important to note the wide confidence bands for this test which are far broader than for any other model discussed in this paper.

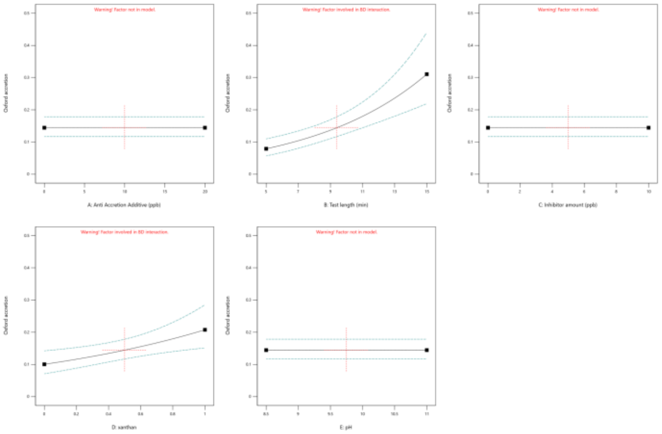


Figure 18. Oxford shale accretion test.

Arne shale accretion test results are shown in Figure 19. Here we see that there appears to be a peak accretion versus time. This could be related to the fact that Arne is a soft shale. Accretion of Arne increases over time but then it starts falling off the accretion tube because it is too soft. Also, worth a note of broad confidence intervals which indicates low repeatability and reproducibility of the test.

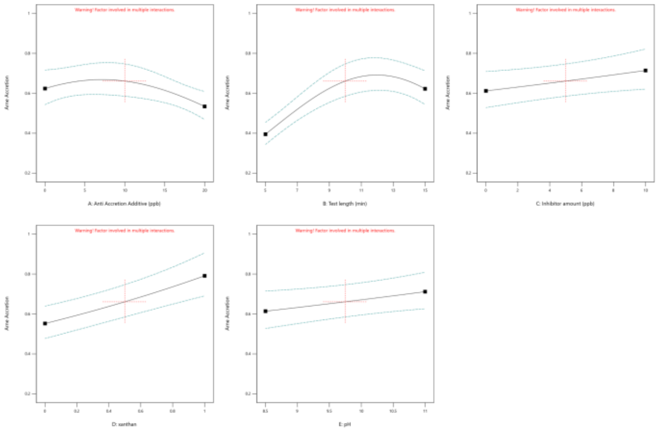


Figure 19. Arne shale accretion test (10-minute test time).

Accretion for ARNE model is highly dynamic which is difficult to report in a paper publication. However, we can compare two scenarios to demonstrate the issues related to current accretion test. Figure 19 shows response from various additives when test is carried out for 10 minutes (maximum accretion) in this scenario we see that low/high dosages of anti-accretion do not contribute much. On the other hand, if the model is set for 5-minute test time (Figure 20) then it appears that anti-accretion is highly beneficial.

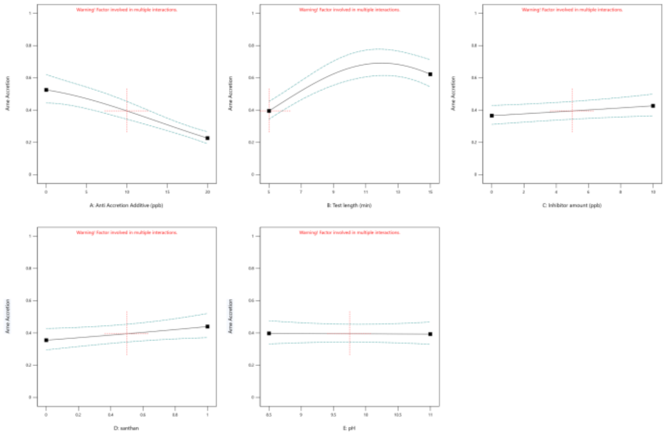


Figure 20. Arne accretion test with 5-minute test time.

Compared either of the Arne accretion results with Oxford accretion results and focusing on anti-accretion additive behavior, conclusions will be dramatically different depending how the test is run. Collectively, this analysis of accretion test shows that results are excessively specific to how the test is conducted. And, for example, changing the accretion test time can result in vastly different conclusion on effectiveness of additives. This suggests limited usability of this test to evaluate anti-accretion products. Furthermore, large confidence intervals indicate high amount of noise in the trends i.e., tests are not very repeatable.

Bulk hardness

Bulk hardness is another method to estimate inhibitive properties of WBM. Cuttings are added to a lab barrel drilling fluid and then recovered after 16-hour hot roll. The cuttings are pressed in an extruder and the force it takes to extrude the cuttings is recorded. Typically, the data is presented as torque reading versus number of turns made as shown in Figure 21. In this chart we see that “no inhibitor” system clearly was inferior as estimated by low torque required to extrude the clay. However, how to compare blue and yellow line, which represent bulk hardness test for 2 different fluid formulations using same shale, is not clear. Is yellow product better because it gives higher torque early on or blue product is better because it reaches higher torque value with less turns. Furthermore, in this comparison only extremes are compared – no inhibitor versus 3 vol. % inhibitor. And, as is typically done, the test does not give information what happens at reduced loadings of inhibitor or what is the repeatability of the test.

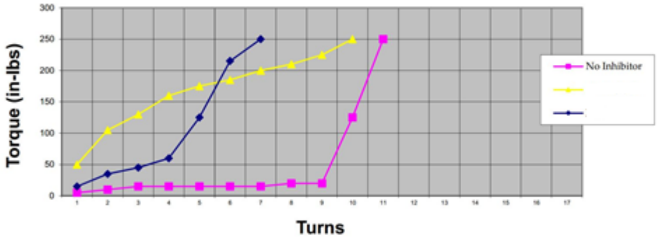


Figure 21. Old way of presenting bulk hardness (from unpublished work).

The number of turns on the bulk hardness meter depend on the amount of shale used for the test and its physical shape. When the piston starts moving, first few turns simply increases shale packing i.e., pieces of shale slide past each other and nothing is extruded. Furthermore, shale section is important to gain sufficient resolution in this test. Shale that is too hard does not differentiate similarly performing inhibitors and can only tell the difference between “no inhibitor” and high loading of high-performing additive. We selected Arne shale as one of the softer and more reactive shales to introduce enough resolution into the test to generate a trend.

For the value of bulk hardness, we observed that for 40 grams of shale it takes approximately 8 turns to extrude all shale and there is a plateau in torque values after piston contacts the shale and until the piston extrudes all the shale; therefore, we selected to record the torque value at 4 turns of the handle to estimate which shale is harder. The variables we selected for this test are shown in Figure 22 and the fluid was formulated at 12 ppg.

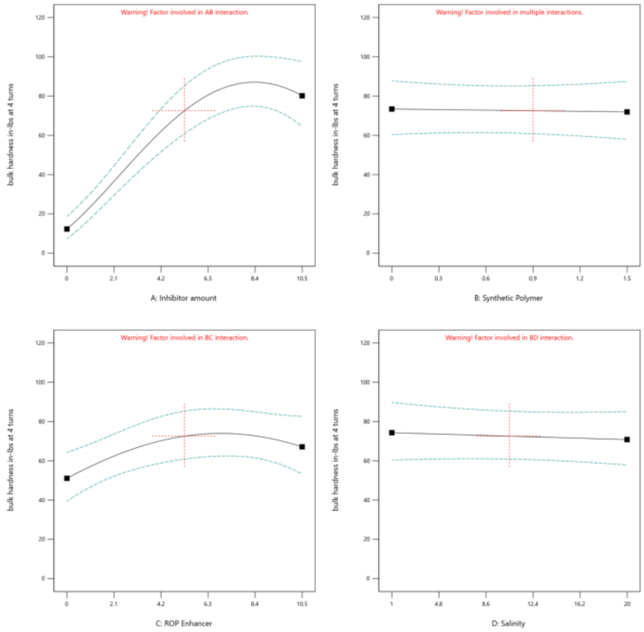
Product	Low Range	High Range
Inhibitor	0 ppb	10.5 ppb
Synthetic Polymer	0 ppb	1.5 ppb
ROP Enhancer	0 ppb	10.5 ppb
Salinity	1 % NaCl	20 % NaCl

Figure 22. Variables for bulk hardness test.

The model we created combines the data from 30 measurements. The model describes the observations reasonably well as seen in model fit statistics (Figure 23).

	R^2	Adjusted R^2	Predicted R^2
Arne bulk hardness	0.9275	0.8946	0.8256

Figure 23. Fit statistics for bulk hardness (the torque value at 4 turns) model.



Bulk hardness model analysis shows that amine inhibitor is the dominating contributor to bulk hardness (Figure 24) and other variables selected in this study do not affect the outcome to a large degree. We also observe that the effect from amine inhibitor on bulk hardness is not linear. Effect on inhibitor concentration on bulk hardness value in Figure 24 shows that bulk hardness increases up to 6 ppb inhibitor and then bulk hardness value remains relatively flat. This observation highlights the importance of using design of experiments for performance analysis. Reaching the maximum benefit for bulk hardness test with less than commonly applied 3 vol. % inhibitor observation correlates to CEC value of the shale and the composition of inhibitor used in this study. Arne shale used in this study has CEC of 11 meq/100g, so the concentration of inhibitor significantly exceeds the available cation-exchange cites on the clay, which is why adding more inhibitor does not provide additional benefit. From the width of confidence intervals, we also see that bulk hardness tests suffers from repeatability issues. Quantification and creation of trends is still possible but requires a lot of data to generate reliable conclusions.

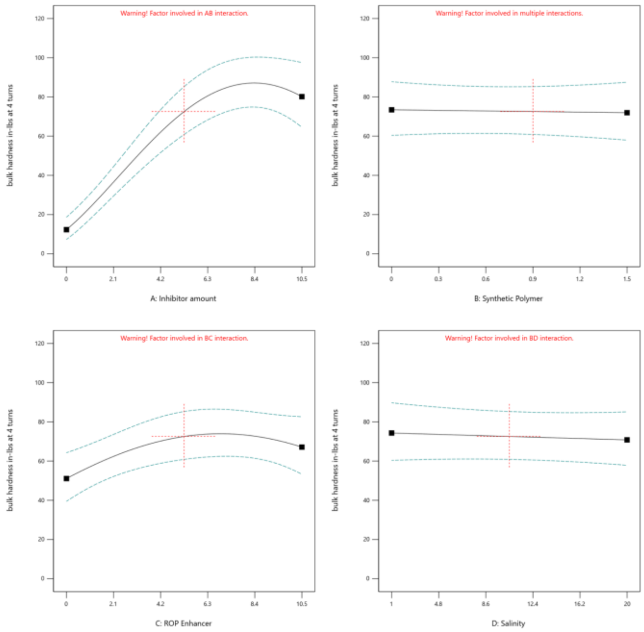


Figure 24. Bulk hardness model analysis.

Bentonite Inhibition Test

Bentonite inhibition test is another test in portfolio of WBM performance testing. Unlike any of the previous examples in this paper, bentonite inhibition test is not performed in a formulated drilling fluid. Typically, the test is performed in water with only inhibitor additive (for high performance WBM) or brine and polymer for polymer WBM, and pure water as blank reference. When conducting a test, large quantities of API bentonite (high CEC) is added to an aqueous solution until viscosity of the slurry increases. Based on the amount of bentonite added before slurry thickens (Figure 25), one estimates how inhibitive the system is (Young et al 2006). The test itself is a viscosity test and as mentioned in this paper, viscosity measurements are highly reliable and repeatable. When looking at the Figure 25 we see that the results for bentonite inhibition test shows large differences between blank, KCl, and polyamine muds; however, the difference between polyamine and new shale inhibitor is not large.

As part of the investigation of what information we gain from various laboratory tests, our interest is to understand if bentonite inhibition test can differentiate performance of amine-based inhibitors. To answer this question, we selected four different commercially available polyamines for evaluation in bentonite inhibition test. Some of these amines are found in highly effective commercial shale inhibitors and some amines are ineffective based on bulk hardness, hydration, or shale dispersion tests.

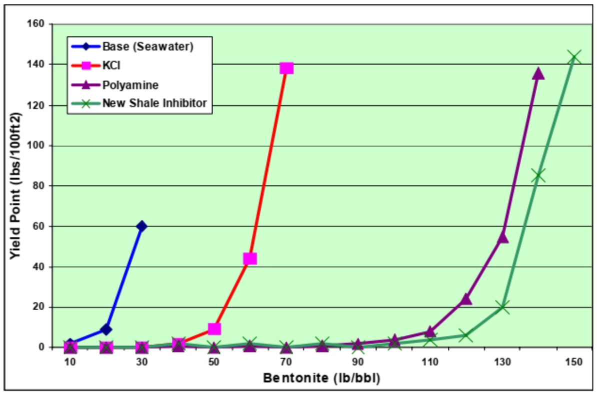


Figure 25. Typical output of a bentonite inhibition test comparing the yield point of three shale inhibitors, 8 lb/bbl and the base fluid (from Young et al 2006).

To make an apples-to-apples comparison that removes factors such as different percent actives in a formulation of a shale inhibitor we are looking at a ratio of inhibited clay per gram of active ingredient (amine). The bentonite inhibition test was performed by the addition of discrete concentrations of bentonite (CEC 49 meq/100 g) powder (from 5 to 40 g with 5 or 10 g increments) to inhibited brine solution containing 50 g of 1% NaCl brine and certain amount of shale inhibitor (**1-4**), that correspond to ~9–44 wt. % clay dispersions. Note that the range of bentonite concentration investigated significantly exceed the typical clay loading found in water-based mud formulations in the field.

Figure 26 plot shows slurry yield point on y-axis (shear stress at 10 s^{-1}) where on x-axis we plot the ratio of clay per gram of amine. Dashed horizonal line was selected as the threshold to a high yield point gel: below the dotted line depicts clay composition with acceptable yield point ($\sigma < 5$ Pa) and above, respectively, as an area of high yield point gel ($\sigma > 5$ Pa). A shear stress of 5 Pa at 10 s^{-1} that is the equivalent of low shear rate viscosity of ~10° at 6 rpm dial readings on a conventional Fann 35 viscometer. This concept approximately corresponds to the turn on the yield point line in old methods of bentonite inhibition test (Figure 25). As the ratio of clay to amine changes (fixed clay, less amine), yield point of the slurry increases. The results in Figure 26 show that when we have excess amine relative to the clay loading (10-15 grams of bentonite per 1 gram of amine) we cannot differentiate amines at all, because all clay-inhibitor dispersions show the equal yield point. In other words, when the ratio of clay to inhibitor is up to 15, all inhibitors show high efficiency. At a higher clay/inhibitor ratio, the yield point of the slurry for amines is significantly different. A similar comparison can be done looking at fixed yield point value, e.g., 5 Pa at 10 s^{-1} (the dotted line in Figure 26) and comparing clay/inhibitor ratios with higher ratio meaning amine inhibits bentonite better.

These results show that bentonite inhibition test is capable of differentiating amines but these results can be misleading. This differentiation of amines does not account for difference in inhibitor formulations. Product 1 with lowest inhibition of clay per gram of amine can be reformulated at higher actives which will improve performance of product 1 in this test. Since formulation details are generally not shared with customers, result from the bentonite inhibition test become difficult to interpret because good result can be obtained from effective inhibitive chemistry or high concentration of marginally effective amine. In another example, product 4 appears to be mid-range in the test can be re-formulated at high actives due to very low cost of the active ingredient but this chemistry has not seen much commercial success due to inadequate performance in other lab tests. Second problem with the bentonite inhibition test is that the test does not correlate with other performance-based tests or field experience. For example, ranking of amines in Figure 26 according to bentonite inhibition test we get **2**>**3**>**4**>**1**. However, same four amines ranked according to bulk hardness test is ranked **2**>**1**>**3**>**4**. And from the field experience, amine **1** is near the top in performance. Bulk hardness and field experience has some correlation and neither of these agrees with the bentonite inhibition test.

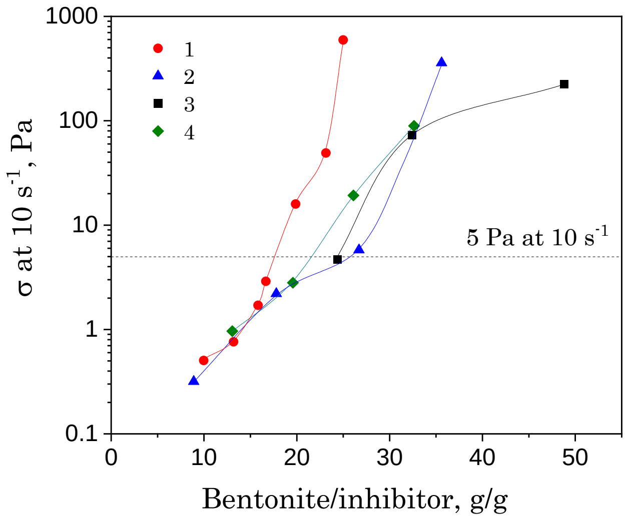


Figure 26. Bentonite inhibition test: yield point vs. ratio of bentonite/inhibitor.

Furthermore, our results in the bentonite inhibition test are normalized for amine content which is not something that is available end users. Contents of active ingredient details is usually not disclosed for commercial products and without knowing the concentration of active ingredient in inhibitor formulation, results from bentonite test cannot be interpreted because we cannot be sure if we are comparing effectiveness of additives or the amount of active ingredient. The distinction between the effectiveness and the amount of inhibitor is important, which we don't get from the bentonite inhibition test, because the concentration of a highly effective additive can be increased to solve the lack of inhibition, but a high concentration of an of ineffective inhibitor will not solve the real problem.

Conclusion

We conclude that for effective and reliable trends, design of experiments methodology should be used. Synergies between products can be observed and more reliable performance trends can be established by improving signal-to-noise with multiple measurements. For repeatability and the type of useful data each test can provide, we are making the following observations:

(1) *6-speed rheology* is reliable and highly repeatable measurement. R600 is ±8% of dial reading. R6 is 5% of dial reading. 10-minute gel is 20% of dial reading.

(2) Shale cuttings integrity tests: *Bulk hardness* test is best differentiator of shale inhibitor performance, and 30% error in measurement is not uncommon. The test responds to quantity of inhibitor in non-linear fashion so for reliable trends multiple measurements at different inhibitor concentrations should be compiled into a single trend to reach a reliable conclusion on performance. *Shale dispersion* is strongly affected by fluid viscosity and results are ±6%. When evaluating performance of products aimed at reducing dispersion, it is imperative that comparison is done in fluids with nearly identical viscosity and same mud weight. *Shale hydration* is affected by salinity of the brine and typical error of ±2%. For typical fluids the signal is larger than the error of measurement occurs at the extreme ranges of the salinity i.e., 0 vs 20 %. Type of salt does not have a significant effect on shale hydration. *Accretion* is poorly reproducible and is a subject to massive variations in related how the test is setup. In the current form it is not a qualitative measurement at best and is generally not a reliable method to quantitate performance of anti-accretion additives.

(3) Linear swell meter test is ±0.8% and only shows strong response to salinity of the system and is poorly sensitive to other additives such as shale inhibitors.

(4) The bentonite inhibition test is based on a viscosity measurement, therefore reliable and reproducible. The test can differentiate amines when normalized for concentration of active ingredient, but results of this test do not always correlate to bulk hardness cuttings integrity test.

(5) When evaluating performance of WBM, there is not a single lab test that will give definitive answer so we recommend that holistic evaluation should be done based on results from multiple tests and performance analysis should be based on enough data points.

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