

# Determining Association of Particles and Emulsion in Invert Emulsion Drilling Fluids: Experiments and Modeling

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This paper was prepared for presentation at the 2015 AADE National Technical Conference and Exhibition held at the Henry B. Gonzalez Convention Center, San Antonio, Texas, April 8-9, 2015. This conference was sponsored by the American Association of Drilling Engineers. The information presented in this paper does not reflect any position, claim or endorsement made or implied by the American Association of Drilling Engineers, their officers or members. Questions concerning the content of this paper should be directed to the individual(s) listed as author(s) of this work.

## Abstract

During various drilling operations (e.g., tripping), drilling fluids can remain in the wellbore from a few hours to several days at static or low-shear conditions. The long-term stability of these fluids is critically important to helping prevent potential emulsion coalescence or separation, particle settling, and sag. Poor stability conditions can present even more risk in complex wells and at high-pressure/high-temperature (HP/HT) conditions, resulting in increased nonproductive time. This paper presents a novel method to quantitatively determine fluid stability.

The associative stability (AS) parameter is introduced and defined, which characterizes the degree of association between suspended solids and the emulsion phase in synthetic and oil-based drilling fluids. AS can be influenced by wellbore conditions, such as temperature, pressure, time, and shear. AS is also influenced by fluid composition (e.g., type of base oil, o/w ratio, type and concentration of emulsifiers and wetting agents as well as the amount, size and type of solids in the system).

Experimental data was used to quantitatively determine AS for several drilling fluid formulations. Static aging tests, using different oil-based fluids, were performed at wellbore conditions. For each of these, various layers of the statically aged mud were analyzed using retort, mud weight, and titration testing. Strong correlation between the AS parameter and sag characteristics of these fluids was observed. Thus, the AS parameter can be used as a direct and meaningful indicator of a fluid's resistance to instability and sag. Additionally, AS can be used to optimize fluid treatments and wellbore operational parameters.

## Introduction

The conventional method of testing the stability of water-in-oil emulsions in drilling fluids is based on using an emulsion tester, which permits application of variable voltage across two electrodes immersed in the emulsion<sup>1</sup>. The voltage is increased until the emulsion breaks, as detected by the surge of current between the electrodes. The voltage necessary for breakdown is considered as a measure of emulsion stability. However, it was observed that this breakdown voltage

depends on several other variables (e.g., mud density, oil/water ratio, emulsifier concentration, and composition<sup>2</sup>). Thus, it was recommended that only trends in electrical stability measurements should be used to make treatment decisions. Therefore, a more meaningful indicator of fluid stability was warranted.

The literature has demonstrated that the presence of micron-size particles in addition to emulsion in drilling fluids enhances fluid stability (i.e., leads to a stronger gel and lower sag tendency<sup>3-5</sup>). Additionally, fluids that have been extensively sheared (e.g., through the bit) have more fluid stability. It can be speculated that the mechanism of particle-droplet interaction plays a major role in determining fluid stability. This paper attempts to quantify this particle-emulsion interaction.

The AS parameter presented in this paper solely quantifies the degree of association between the particle and emulsion phases; thus, it is direct, and therefore a better indicator of drilling fluid stability (e.g., resistance to sag). The absolute values as well as trends of AS described can be used to help make fluid-treatment decisions.

## Theory: Association Stability

The volume fractions of mud components that include oil, brine, low gravity solids (LGS), and barite are denoted respectively as:

$$\phi_{oil}, \phi_{brine}, \phi_{LGS}, \phi_{barite}$$

For a given mud sample, these fractions were determined by performing standard component-wise mass balance on the data obtained from retort, titration, and mud-weight testing.

For the initial uniform mud, the fractions are denoted as:

$$\phi_{oil}^i, \phi_{brine}^i, \phi_{LGS}^i, \phi_{barite}^i$$

On the contrary, for the sagged fluid bottom section, after static aging (**Figure 1**), the fractions in the fluid are denoted as:

$$\phi_{oil}^b, \phi_{brine}^b, \phi_{LGS}^b, \phi_{barite}^b$$

In the present method, the AS is considered to be dependent on degree of association between the particle and emulsion phases (i.e., whether solids (specifically barite) and brine phase settle together as described by Eq. 1).

$$AS = \frac{\phi_{\text{barite}}^i / \phi_{\text{brine}}^i}{\phi_{\text{barite}}^b / \phi_{\text{brine}}^b} \times 100\% \quad (1)$$

The range of AS is denoted by  $0 \leq AS \leq 100\%$ . For the drilling fluid,  $AS \approx 0$  indicates a fully unstable association system, while  $AS \approx 100\%$  represents a fully stable association between the particle and emulsion phases, as shown in **Figure 1**.

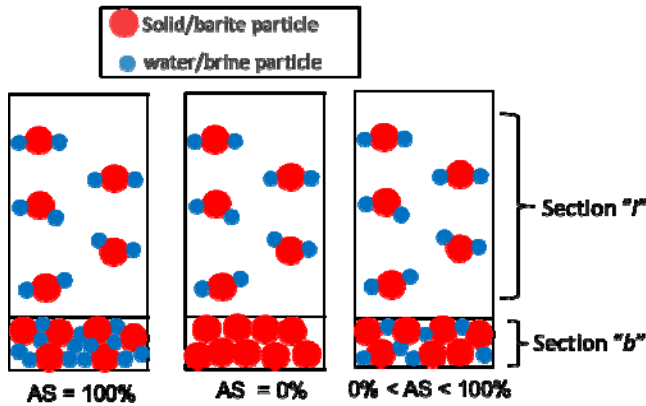


Figure 1: Schematic of the concept of AS.

Alternative methods for measuring AS can also include replacing the barite fraction in Eq. 1 with the total amount of solids and/or replacing the brine fraction with the fraction of water present in the fluid.

The AS could be direct and meaningful indicator of the fluid stability (e.g., quality of sagged portion) and correspondingly could be used when making fluid-treatment decisions.

### Experimental Study: Materials

Several organoclay-free invert emulsion oil-based drilling fluids were prepared; the fluids were hot-rolled at the testing temperature for 16 hours before performing the following tests. **Appendix I** presents examples of the selected drilling fluids (Fluids A and B) and their composition.

### Experimental Methodology

**Static Aging Testing:** Standard static aging testing was performed on the selected invert-emulsion oil-based muds (OBMs) at various temperatures and under 100 psi pressure. A petri-dish container (35-cc capacity) was placed at the bottom of the aging cell to collect the settled (or sagged) mud.

**Retort Testing using API Recommended Practice 13B-2, Section 8:** Retort testing was performed on the selected OBMs. For each OBM, two retort tests were performed, first on the fresh initial mud with uniform composition, and

secondly on mud collected in the petri-dish at the bottom of static aging cell.

**Mud-Weight Testing:** Mud-weight (fluid density) measurements were performed on the selected OBM. For each OBM, two mud-weight tests were performed, first on the fresh initial mud with uniform composition, and secondly on mud collected in the petri-dish at the bottom of static aging cell.

**Chemical Analysis (Titration) Testing using API Recommended Practice 13B-2, Section 9:** Chemical analysis (titration) tests were performed on the selected OBMs to determine the salt type and concentration in the drilling fluid. Generally, because the salt concentration in water is not expected to change during aging, this test does not need to be performed on the bottom mud section in the aging cell.

## Results and Analysis

### Effects of Temperature on AS

The formulated Fluid A was tested at various temperatures to investigate the dependence of AS on temperature (**Table 1**).

Table 1: AS of drilling Fluid A after static aging of 48 hours at various temperatures.

(Properties of Fluid A: o/w = 80/20 MW = 12 ppg,

$$\phi_{\text{brine}}^i = 0.17, \phi_{\text{barite}}^i = 0.14, \phi_{\text{barite}}^i / \phi_{\text{brine}}^i = 0.82.)$$

Drilling Fluid A	$\phi_{\text{brine}}^b$	$\phi_{\text{barite}}^b$	$\frac{\phi_{\text{barite}}^b}{\phi_{\text{brine}}^b}$	AS (Using Eq. 1)
Temperature (T) = 150°F	0.26	0.22	0.85	97%
Temperature (T) = 250°F	0.31	0.34	1.1	77%

After conditioning, retort, mud-weight and titration tests were conducted on the initially uniform drilling fluid. Using the test data, component-wise mass balance calculations were performed to estimate volume fractions of the fluid components that include oil, brine, and LGS in the initially uniform fluid. The corresponding fractions  $\phi_{\text{oil}}^i, \phi_{\text{brine}}^i, \phi_{\text{LGS}}^i, \phi_{\text{barite}}^i$  are indicated in Table 1. Afterward, the uniform Fluid A was kept for static aging of 48 hours at 150°F aging temperature. A petri-dish container was placed at the bottom of the aging cell. The settled (or sagged) portion of the fluid at bottom of the static aging cell was collected in the petri-dish and then retort, mud weight, and titration tests were conducted on this sample in the petri-dish. Using the test data, component-wise mass balance calculations were performed to estimate volume fractions of the fluid components that include oil, brine, and LGS in the sagged portion of the fluid at 150°F. The corresponding fractions of the fluid components at 150°F are indicated in Table 1.

The same tests described were repeated by placing Fluid A for static aging at a varying temperature, 250°F. The corresponding fractions of the fluid components in the sagged fluid at 250 °F are indicated in Table 1.

Analysis of the Table 1 data evidently shows that the AS

(as determined using Eq. 1) decreases with temperature from 97 to 77%, which is intuitive because the additive's (emulsifier/wetting agents) performance is generally observed to be weakened with increasing temperature.

### Effect of Ageing Duration on AS

The formulated Fluid B was tested at varying temperatures to investigate dependence of AS on duration of ageing (Table 2).

**Table 2: AS of drilling Fluid B after static aging at 250°F for various durations of ageing.**

(Properties of Fluid B: o/w = 80/20 MW = 14.5 ppg,

$$\phi_{\text{brine}}^i = 0.16, \phi_{\text{barite}}^i = 0.22, \phi_{\text{barite}}^i / \phi_{\text{brine}}^i = 1.37.)$$

Drilling Fluid B	$\phi_{\text{brine}}^b$	$\phi_{\text{barite}}^b$	$\frac{\phi_{\text{barite}}^b}{\phi_{\text{brine}}^b}$	AS (Using Eq.1)
Ageing duration = 24 hours	0.24	0.36	1.5	91%
Ageing duration = 96 hours	0.24	0.39	1.62	84%

After conditioning, retort, mud-weight, and titration tests were conducted on the initially uniform drilling fluid. Using the test data, as described previously, the volume fractions of the initially uniform Fluid B  $\phi_{\text{oil}}^i, \phi_{\text{brine}}^i, \phi_{\text{LGS}}^i, \phi_{\text{barite}}^i$  were determined, as indicated in Table 2.

Afterward, the uniform Fluid B was kept for static aging at 250°F for an aging duration of 24 hours. The settled portion of the fluid at bottom of the static aging cell was collected in the petri-dish and then retort, mud-weight, and titration tests were conducted on this sample in the petri-dish. Using the test data, as described previously, the volume fractions of the fluid components in the sagged portion of the fluid were determined. The corresponding fractions of the fluid components for the ageing duration of 24 hours are indicated in Table 2.

The same tests described previously were repeated by placing Fluid B for static aging for a longer duration of ageing, 96 hours (at the same ageing temperature). The corresponding volume fractions of the fluid components in the sagged fluid are indicated in Table 2.

The analysis of the Table 2 data evidently shows that the AS (as determined using Eq. 1) decreases with duration of the ageing. It indicates the amount and quality of the additives (emulsifier/wetting agents) should be adjusted based on the adversity of operational parameters (i.e., for example, how long the fluid will be left uncirculated in the wellbore during tripping operations).

### Relation of AS to Sag

The presented quantitative information on AS can be used determine how it affects the sag response of the fluid in terms of composition of the sagged fluid as the particulate phase settles (Table 3).

**Table 3: Effect of AS on sag demonstrated using data from Fluid A.**

Drilling Fluid A	AS (Using Eq.1)	$MW^b$ (Sagged Fluid Mud Weight from Aging Test)	$MW^{AS=100\%}$ (Calculated Considering 100% AS)
Temperature (T) = 150°F	97%	13.8	13.6
Temperature (T) = 250°F	77%	17.5	16.2

The experimentally measured mud weight of the sagged portion of the fluid, which was collected in the petri-dish at the end of static ageing testing, is denoted as  $MW^b$ . In this context,  $MW^{AS=100\%}$  represents theoretically calculated mud weight of the sagged portion of fluid assuming AS = 100%, where a strong association of barite to brine helps prevent excessive accumulation of barite in the sagged fluid.

The experimentally measured mud weight of the sagged fluid,  $MW^b$ , would be generally higher than  $MW^{AS=100\%}$ . This is because, in most cases, AS < 100%. Additionally, the lower the values of AS, the greater accumulation of barite in the sagged fluid, which would further increase  $MW^b$ , beyond the theoretically calculated sagged fluid mud weight ( $MW^{AS=100\%}$ ). The condition of  $MW^b \gg MW^{AS=100\%}$  would be a concern in the field because it would be difficult to remix.

Table 3 demonstrates the effect of AS on sag based on the data obtained from Fluid A. At  $T = 150^\circ\text{F}$ , the fluid shows excellent AS (AS = 97%). The experimentally measured mud weight of the sagged portion of the fluid was  $MW^b \approx 13.8$  ppg, while the theoretically calculated mud weight of the sagged of fluid (assuming 100 % AS) was  $MW^{AS=100\%} \approx 13.6$ . The corresponding difference would be  $[MW^b - MW^{AS=100\%}] \approx 0.2$  ppg; this smaller differential indicates that the sag response was not aggravated because barite-emulsion association remained strong.

On the contrary, Fluid A at  $T = 250^\circ\text{F}$  shows lower AS, AS = 77%. As shown in Table 3, the experimentally measured mud weight of the sagged portion of the fluid was  $MW^b \approx 17.5$  ppg, while theoretically calculated mud weight of the sagged of fluid (assuming 100 % AS) was  $MW^{AS=100\%} \approx 16.2$ . The corresponding difference would be  $[MW^b - MW^{AS=100\%}] \approx 1.3$  ppg, which is significantly higher (compared to lower temperature), indicating that the sag has worsened because of weaker association of barite-emulsion at this temperature; hence, there could be a need for a better additives (emulsifier/wetting agents) package.

Thus, the information on AS could be used to quantitatively determine its affect on the sag response of the fluid, and corresponding corrective action could be taken. Similarly, the effect of pressure, aging duration, applied shear condition, as well as the fluid composition on AS could be investigated. Ultimately, a critical value of AS could be

determined that could act as one of the acceptability criteria for the fluid's performance under given conditions.

### Conclusions

- AS experimentally measures the degree of association between the solids and emulsion phases in a field.
- AS would depend on the type of fluid and operating conditions, such as temperature and ageing duration.
- AS could be used as a direct and meaningful indicator of fluid stability and associated behaviors (e.g., sag response) of the fluid.
- The AS value of the fluid can act as one of the acceptability criteria for the fluid's performance under given conditions and could be used for making fluid-treatment decisions.

### Acknowledgments

The authors thank Halliburton management for permission to present this work.

### Nomenclature

*OBM* = oil based mud

*ppg* = pounds per gallon

*o/w* = oil water ratio

*LGS* = low gravity solids

*AS* = associative stability

$MW^b$  = experimentally measured mud weight of sagged fluid

$MW^{AS=100\%}$  = theoretically calculated mud weight of the sagged portion of fluid assuming  $AS = 100\%$

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### Appendix 1: Fluid Formulations

#### Fluid A (o/w = 80/20, MW = 12 ppg)

Base fluid (bbl)	As required
Emulsifier (ppb)	8
Lime (ppb)	1.5
Filtration control agent (ppb)	2.5
CaCl <sub>2</sub> brine, (200 K)	As required
LGS I (ppb)	5
LGS II (ppb)	20
LGS III (ppb)	20
Barite (ppb)	As required
Viscosifier (ppb)	3

#### Fluid B (o/w = 80/20, MW = 14.5 ppg)

Base fluid (bbl)	As required
Emulsifier (ppb)	8
Lime (ppb)	1.5
Filtration control agent (ppb)	2.5
CaCl <sub>2</sub> brine, (200 K)	As required
LGS I (ppb)	5
LGS II (ppb)	20
LGS III (ppb)	20
Barite (ppb)	As required
Viscosifier (ppb)	3