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Designing Laboratory Test Protocols for Asphaltenes Deposition

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ABSTRACT

Asphaltenes deposition is associated with several detrimental effects on oil production, paramount among these being production deferment and integrity issues. The current paper presents an investigation of asphaltenes risk assessment and mitigation strategies using a dynamic flow test method. It includes a study of the key parameters that influence observation of asphaltenes deposition in the laboratory and how laboratory testing methodologies can be designed to match more closely the processes occurring in the field.

In these dynamic tests, dead crude oil is co-injected with *n*-heptane through a capillary and an inline filter, allowing assessment of both deposition *and* precipitation in a single test. The method is able to assess deposition tendency and rank asphaltenes inhibitor chemicals under flowing conditions and at more field-representative temperature and pressure, and with much lower volume fractions of *n*-heptane than required for conventional asphaltenes laboratory tests.

Fluid dynamics is recognised to play a key role in the extent of asphaltenes deposition and is often most severe under low-shear conditions because erosive forces can reduce or eliminate deposition at higher shear. Using the dynamic flow test method with capillaries of differing internal diameter, we investigate the factors that either promote or suppress deposition, and determine certain boundary conditions where deposition is inhibited under the influence of increasing shear.

The dynamic method is also used to evaluate the effectiveness of asphaltenes inhibitor chemicals against both precipitation *and* deposition, thus demonstrating that this approach provides much more relevant chemical-performance data than conventional test approaches, and may even elucidate why chemicals selected using non-dynamic methods often perform indifferently in the field or sometimes fail to perform completely.

Key words: Asphaltenes, Asphaltenes Inhibitor, Screening, Deposition, Precipitation, Inhibitor Qualification, Dynamic Flow Test, Fluid Dynamics

INTRODUCTION

Background

It is estimated that approximately 5 – 10% of the world's crude oils are susceptible to asphaltenes issues, which can cause flow restrictions, compromised integrity including malfunction of control and monitoring equipment, loss of well access for interventions, enhanced emulsion stability and internal under-deposit corrosion. Despite this, currently used methods to assess asphaltenes deposition risk and evaluate the laboratory performance of asphaltenes inhibitors are non-optimal because the conditions used are so far from those prevailing in the field. This can lead to inadequate assessment of risk, erroneous ranking of candidate inhibitors and even selection of chemicals that may not be beneficial in the field at all.

There are many techniques for evaluating asphaltenes risk.¹⁻⁶ However, the most prevalent techniques are only useful for determining when asphaltenes may *precipitate* but not when they may *deposit*. Although asphaltenes precipitation can lead to certain operational challenges (e.g., enhanced emulsion stability, solids fill in separators), these generally occur in surface production facilities and do not result in flow-assurance or integrity problems further upstream. Asphaltenes deposition is a complex process that depends on fluid turbulence, wall shear stress, surface type and characteristics, particle size of the precipitated flocs and particle-surface interactions, as well as the primary factors affecting precipitation (temperature, pressure and fluid composition).⁷

There are reports in the literature of capillary loop tests for the investigation of asphaltenes deposition.⁸⁻¹² Most of these have been conducted at temperatures and pressures close to ambient. Pressure plays a key role in asphaltenes deposition tendency so it would appear sensible to assess laboratory asphaltenes deposition tendency at the conditions under which deposition normally occurs in the field (typically, elevated T, P). Furthermore, many of these tests were conducted using glass capillaries (to allow visual observation) rather than those composed of steel, the most-likely material used for production tubing in the field.

In this paper we present a new laboratory test method that is relatively quick, inexpensive, based on deposition and offers good correlation with field behaviour. The technique can be used both to assess asphaltenes deposition risk and to evaluate asphaltenes inhibitor (AI) chemical efficiency at preventing deposition and precipitation. The asphaltenes deposition flow test involves the mixing of crude oil with a precipitant solvent (typically, *n*-heptane, though the method can also be used for determining the effect of mixing with a field condensate) in a continuous injection system. The addition of solvent to destabilize asphaltenes is designed to mimic the field destabilization mechanism, which for asphaltenes “self scaling” (that which occurs primarily as a result of pressure reduction on a single fluid stream) occurs by the volumetric expansion of the lighter, more compressible paraffinic ends as pressure reduces in the production system. The mixed fluids then pass through a steel microbore capillary and sintered metal filter in series. A schematic of the system is presented in Figure 1. The current apparatus can be heated to 180°C and pressurized to 345 bar. Differential pressure (dP) is recorded across each component. An increase in dP during a flow test indicates an increasing flow constriction in that component: across the capillary this indicates asphaltenes deposition therein, while across the filter indicates plugging by precipitated asphaltenes that did not deposit in the capillary upstream. An example data plot is presented in Figure 2, illustrating both deposition and precipitation in a single test.

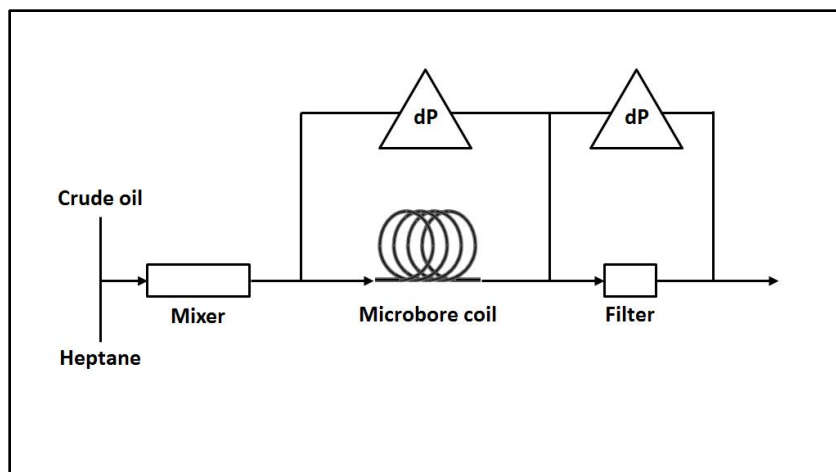


Figure 1: Schematic of the asphaltenes deposition flow rig.

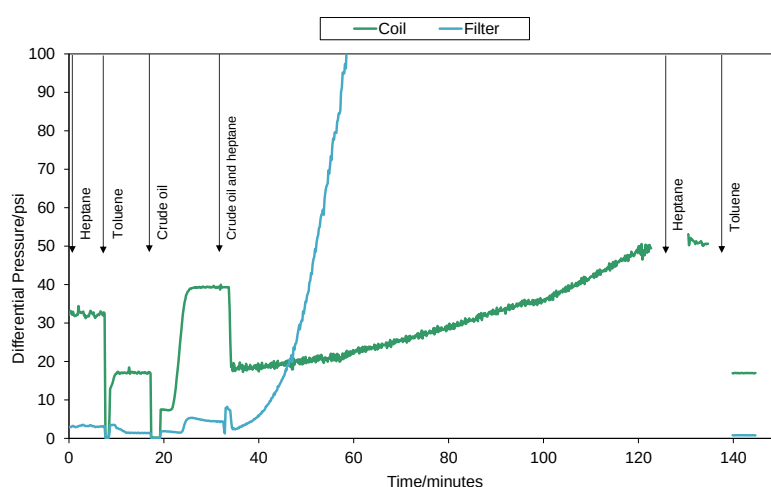


Figure 2: Asphaltenes deposition test example plot showing progressive deposition in the steel capillary and plugging of the filter.

Parameters Affecting Asphaltenes Deposition

A number of parameters may be varied when designing test conditions to represent field conditions most closely. A non-exhaustive list is presented below, including considerations to be made when selecting each value.

Temperature and Pressure: Typically chosen to match values expected within the asphaltenes precipitation envelope (either measured or modelled).

Capillary length: In the apparatus reported here, a length of 18 m has been found to fulfil the following criteria:

- It allows sufficient residence time for asphaltenes precipitation to occur when the linear flow velocities are typical of nominal liquid rates in the field
- The degree of deposition that can occur within this time and over this length generates a readily measurable increase in dP

Capillary diameter: Besides also affecting both the residence time and the incremental dP due to asphaltenes deposition, the internal diameter of the capillary has a significant effect on the fluid dynamics of the system. This is discussed in more detail below.

Flow rate: The flow rate also affects the fluid dynamics of the system. This is discussed in more detail below.

Filter pore size: Altering the filter pore size will alter the minimum particle size required to plug the filter. For example, if a 60 micron filter element is used, significant precipitation may occur but would likely go undetected. On the other hand, if a 0.5 micron filter element is used then the filter may plug too quickly, even with only mild degrees of precipitation (or even due to residual formation and/or drilling fines present in the oil sample). Our experience has shown that a pore size of 5 – 20 micron allows essentially exclusive observation of precipitation that is induced by the mixing of crude oil and the precipitant solvent. Moreover it does so at a rate that is high enough to be readily measured but not so high that it plugs before a readily measurable increase in dP can be determined for the capillary.

Identity of precipitant: The most representative precipitant is likely to be a mixture of light *n*-alkanes, such that its addition will bring the composition of the mixture close to that which exists in live oil close to the bubble point. However, this is operationally challenging and very complex to achieve, and so it is much simpler to use a single precipitant solvent that is liquid at ambient temperature/pressure. The identity of the precipitant can be chosen based on the average solvency characteristics of the combined *n*-alkanes; typically *n*-pentane, *n*-hexane or *n*-heptane are found to be good matches.

Crude oil / precipitant ratio: The ratio between crude oil and precipitant should be selected to most closely represent the characteristics of the fluid close to the bubble point. In some cases it may be necessary to increase the proportion of precipitant above this criterion by a small degree, making the asphaltenes supersaturation slightly more severe, in order to observe deposition within reasonable timescales in the laboratory.

Fluid Dynamics

As noted above, the fluid dynamics of the system can be influenced by altering both capillary diameter and flow rate. Fluid dynamics are of particular interest when assessing asphaltenes deposition (as opposed to precipitation only), with a number of literature reports suggesting that deposition may be limited or even halted under certain high-shear regimes.¹²⁻¹⁴ Critical shear rates are dependent on the asphaltenes nature and stability state and are, therefore, specific to each crude and production system, with reported values varying from 1,000 s⁻¹ to 100,000 s⁻¹. In order to design a test methodology that allows assessment of asphaltenes deposition risk and evaluation of inhibitor performance, it is critically important to select appropriate hydrodynamic conditions, *i.e.*, those that most closely match those prevailing in the field.

Scope of the Current Work

Laboratory tests were conducted to assess the effects of altering fluid dynamics on asphaltenes deposition. Additionally, the typical workflow for assessing asphaltenes inhibitor (AI) chemical performance is described using a case study.

EXPERIMENTAL PROCEDURE

Asphaltenes Dispersancy Tests

The asphaltenes dispersancy test (ADT) is one of the most common methods for screening AI, due to its speed, ease and low cost.⁵ ADT is conducted using a large excess of *n*-heptane (most commonly) in order to achieve measurable asphaltenes precipitation and to make the resulting solution sufficiently

transparent to observe the solids. As such, the technique favours AI that reduce either the extent or the rate of particle flocculation, maintaining the floc size below that which is “visible” to the technique. It does not provide any information on deposition tendency. Therefore, although ADT can be used to evaluate whether an AI chemical is effective at preventing asphaltenes precipitation, it does not necessarily correlate with its ability to prevent deposition in field pipelines and equipment under flowing conditions.

The ADT method was used in the present work in order to provide a comparison for the AIs tested using the flow-loop deposition test method, *i.e.*, effectiveness in preventing precipitation *versus* effectiveness in preventing deposition. First, samples of crude oil (ranging from 50 to 300 μ l) were dosed into 12.5 ml *n*-heptane in graduated tubes. The tubes were shaken and left to stand for one hour at ambient temperature. The quantity of oil that gave between 4 and 10% sedimentation by volume (of the oil/heptane mixture) in the blanks (without AI addition) was chosen for chemical screening tests.

Two AI formulations, AI-A and AI-B, were tested with Crude A using both ADT and the asphaltenes deposition test method. Each AI was dosed directly into the crude oil; concentrations are quoted vol/vol with respect to the oil. The dosed oil was then heated at 60°C for a period of 10 minutes prior to testing. The selected amount of dosed crude oil was then added to 12.5 ml *n*-heptane in graduated tubes. The tubes were then shaken and left to stand for one hour at ambient temperature and the volume of sediment was recorded.

Asphaltenes Deposition Flow Tests

Crude oil and *n*-heptane were co-injected into the flow system and passed through a passive mixer to ensure complete mixing. The combined fluids then flowed through a steel microbore capillary and a sintered metal filter in series. The differential pressures across the mixer, capillary and filter were measured, allowing assessment of any deposit or blockages arising from solid deposition or precipitation. Experiments were carried out with two crude oils (termed Crude A and Crude B). Crude A was used in a typical inhibitor screening workflow. Crude B was used to investigate the effects of modifying fluid dynamics on the rates of deposition and precipitation.

Inhibitor Screening Workflow

When beginning flow-loop testing for a particular crude oil, optimization tests are first carried out in order to correlate the laboratory test with the field system. This includes defining a suitably representative temperature and pressure, as well as ensuring that capillary length, internal diameter, filter pore size, flow rate and crude oil / precipitant solvent ratio have all been selected for the particular oil under test to show suitable degrees of precipitation and deposition within the apparatus. These factors play a critical role in the extent of asphaltenes precipitation and deposition on the capillary wall, and they are tuned to match field conditions and (if available) field observations. Typically, the final parameter to be tuned is the ratio of crude oil to precipitant solvent used in the test, as this is perhaps the strongest determinant of the asphaltenes supersaturation, and hence, the extent and rate of precipitation and deposition.

Crude A was reported to exhibit asphaltenes deposition within the downhole production tubing in the field. Therefore, optimisation tests were carried out in order to produce asphaltenes deposition within the capillary under a temperature and pressure that was representative of the field. Capillary dimensions and flow rate were chosen to match typical linear velocities during production (the system can achieve velocities ranging from 0.01 to 2 m/s). The ratio of crude oil and *n*-heptane was varied until suitable rates of increase in dP in the capillary (deposition) and filter (precipitation) were recorded (typically an increase of 2 – 4 psi within 90 minutes).

Once tuned conditions were optimized, deposition tests were conducted with the two AI chemicals added. Each AI was dosed into Crude A at several concentrations (each quoted with respect to the oil volume before addition of *n*-heptane).