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IMPACT OF MIXING ON CRUDE OIL FOULING TESTS

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ABSTRACT

For many years, crude oil fouling studies have used different types of test units, especially those appropriate for annular and tubular flow, as well as stirred batch reactor rigs. However, in these tests, limited attention has been given to what occurs before an experiment reaches steady-state conditions. This paper focuses on crude oil mixing before the fluid is loaded into the test apparatus and the impact of that mixing on the resulting fouling data. Tests show that different mixing methods can produce different heat transfer coefficients and different fouling rates.

INTRODUCTION

Crude oil is a very complex fluid composed of a seemingly infinite number of molecules. One particular metric in the analysis of crude oils is the solids content, which varies widely (Watkinson, 2005). Solids can originate from a large range of sources in crudes, some of these insoluble [such as silt and corrosion products (ESDU, 2005)] and some that under certain conditions resolubilize or precipitate out [for example, gums, asphaltenes, and waxes (Watkinson et al., 2000)].

Most insoluble solids have a greater density than the liquid, and, if left undisturbed, they naturally settle to the bottom of the liquid. The result is that samples taken from the top and bottom of a crude oil barrel have different particulate content. If a barrel of oil is stored for a significant amount of time, these particulates could stick to the wall of a barrel, or even start reacting with and corroding the barrel, as Watkinson et al. (2000) observed.

Because the particulates in oils are often thought to be some of the key contributors to fouling, variability in solids content between samples jeopardizes the accuracy and repeatability of fouling data. It is therefore critical that, when a single crude oil is tested multiple times and at a variety of conditions, the fluid composition is consistent.

HTRI fouling researchers (Bennett et al. 2006) have long recognized the need for thorough mixing of a crude oil sample prior to testing. However, the methods implemented have changed over the years, increasing the need to identify what effects poor mixing can have on the resulting measured fouling rates. Recent fouling research at HTRI has involved the testing of self-incompatible crude oils that contain insoluble asphaltenes at room temperature. Concerns that asphaltenes in the crude oil may settle out to the bottom of the barrels before a sample is collected for testing has led HTRI to reconsider its method to mix crude oil in the drum.

Fig. 1 shows what the crude looks like when inspected using a microscope to illustrate the extent of the insoluble asphaltenes present at room temperature.

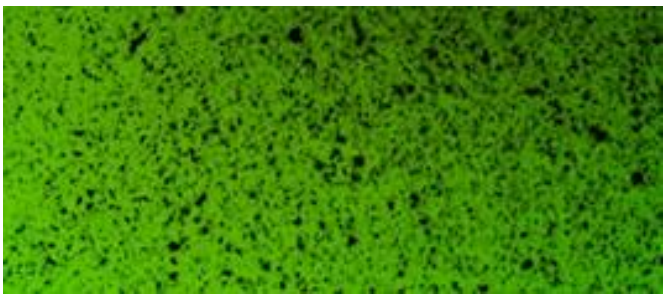

Fig. 1 Microscopic image (40× magnification) of crude oil used in mixing experiments conducted in the RFU

METHODS

Crude Oil Mixing

HTRI stores barrels of crude oil in a warehouse until testing commences. Testing of a crude oil may begin the day after it is received, or it could occur several months or years later. Over extended periods of time, sediments settle out of the crude oil, tending to stick to the bottom and the sides of the barrel.

Fig. 2 provides a visual representation of a well-mixed barrel of crude oil and an unmixed barrel of crude oil and shows the natural tendency of heavy particles to accumulate on the bottom and sides of a barrel when left unmixed.

Therefore, it is important to thoroughly mix the crude oil so that each test uses a crude oil with a comparable composition.

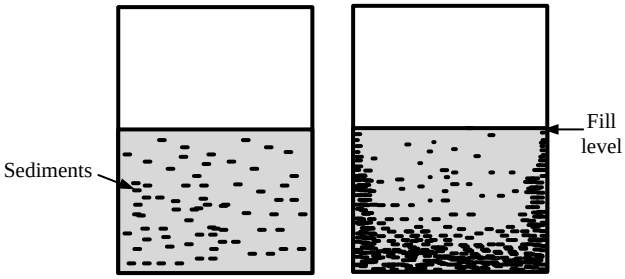

Fig. 2 Visual comparison of a well-mixed crude oil to an unmixed crude oil

Previous method of mixing crude oil. HTRI previously mixed a crude oil using a rotary hand pump. A technician manually recirculated the crude into the barrel for approximately 15 minutes. Not only was this method tedious, but because the method relied on different pump operators, results were inconsistent. Fig. 3 shows a schematic of the setup for manually mixing crudes with a drum pump.

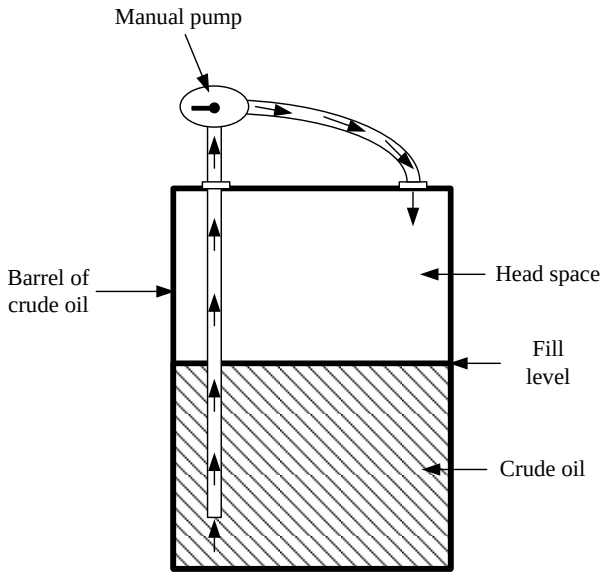

Fig. 3 Diagram of old HTRI mixing method for a barrel of crude oil

To see how well this method mixed the crude oil, a comparison was performed. Before any mixing occurred, the barrel was laid on its side, and a picture was taken through the bung hole to see if anything had adhered to the walls of the barrel. This picture, as well as images of the inside of the barrel following different methods of mixing, is shown in Fig. 4. The bottom and sides of the barrel where the crude oil had been were extremely dark and covered by sediment.

The crude oil was then mixed with the manual pump, as described earlier. Again, a photo was taken of the crude oil in the barrel. The second image in Fig. 4 shows a slight difference but little actual improvement.

The last test was to manually roll the barrel on its side for 15 minutes in an attempt to better mix the fluid and make sure that any sediment was well mixed in the crude oil. In the third image in Fig. 4, the bottom of the barrel no longer has particulates adhered to it, and only residual oil is observed; thus, it can be concluded that the crude oil was better mixed after the barrel was rolled.

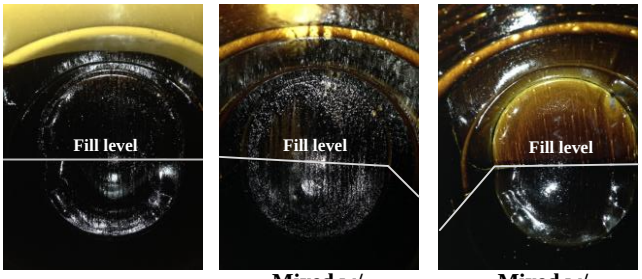

Fig. 4 Visual comparison of three different mixing methods

This comparison shows significant improvement to mixing when the barrel was rolled around manually than when a rotary hand pump was used, suggesting that a new method is necessary to better mix the sediments into the crude oil.

New method of mixing crude oil. As mentioned previously, rolling a barrel of crude oil was an effective way to mix the crude oil. However, manually rolling a barrel is tedious, and human error and inconsistency can again adversely affect the mixing. HTRI therefore opted to invest in a drum tumbler to better mix crude oil. Fig. 5 shows the drum tumbler rotating a barrel of crude oil.

Fig. 5 Drum tumbler acquired by HTRI to mix crude oils

The crude oil barrel is strapped into the barrel tumbler, and an arm from the drum tumbler lifts the barrel and tilts it before rotating it. The new practice at HTRI is to set the drum tumbler to its maximum speed setting (20 rpm) and mix a barrel for 15 minutes at full at a 45-degree angle before using the crude oil in a fouling test. This procedure allows the crude oil to be consistently mixed before each test.

The Rotating Fouling Unit (RFU)

All fouling tests detailed in this paper were conducted using the RFU, a batch unit designed and built by HTRI to conduct fouling tests using a small volume (2.8 L) over a short period of time (< 24 hours). Lane (2013) describes the original design of the unit in more detail. A schematic of the RFU is shown in Fig. 6. Fouling deposits are collected along a heated section of the probe.

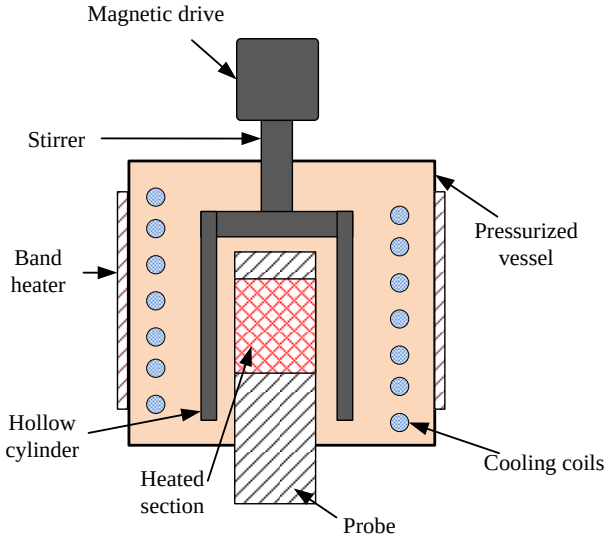

Fig. 6 Schematic of the design of the RFU

The Use of Nusselt Numbers to Distinguish Composition Differences between Various Batches of the Same Fluid

As discussed by Harris et al. (2015), the expected Nusselt number of a crude oil in the RFU can be predicted using Eq. (1).

$$\mathrm{Nu}_{pred} = a\,\mathrm{Re}^{b}\left(\mathrm{Pr}^{c}\left(\frac{\mu_b}{\mu_w}\right)^{d}\right) \quad (1)$$

This equation was derived from a series of tests conducted at HTRI with p-xylene and Duratherm as the test fluids. During an experiment, the Nusselt number is calculated using Eqs. (2).

$$\mathrm{Nu}_{\exp} = \frac{h(D)}{k_f} \quad (2)$$

where

$$h = \frac{Q_p}{A\left(T_w - T_b\right)} \quad (3)$$

The ratio of the experimental Nusselt number to the predicted Nusselt number is referred to as the Nusselt ratio and can be used to assess the extent of boiling, with a Nusselt ratio close to 1 (< 1.25) indicating single-phase heat transfer. The Nu ratio is shown in Eq. (4).

$$\text{Nu ratio} = \frac{\mathrm{Nu}_{exp}}{\mathrm{Nu}_{pred}} \quad (4)$$

Comparing the Nusselt number obtained from an experiment to the expected Nusselt number obtained using Eq. (4) is a valid way to determine when two-phase heat transfer is occurring. The Nusselt ratio can also be used to indicate how well-mixed the crude is and how concentrated the fluid is with particulates.

EXPERIMENTAL SUMMARY

Four fouling runs (A-D) were performed in the RFU to determine the effectiveness of mixing a barrel of crude oil with the hand pump (A and B) or the drum tumbler (C and D). The following conditions were maintained constant during each run:

- T_b = 226 °C
- Q_p = 264 W
- P = 53-58 bar (5.3-5.8 MPa)
- ω_o = 500 rpm

The runs were performed in pairs to check the repeatability and consistency of the mixing methods. If the methods are repeatable, then similar fouling data would be expected for identical conditions. The differences in the results of the experiment are explained in greater detail in the next section.

RESULTS AND DISCUSSION

The recorded test conditions, fouling rate, and Nu ratio results from each run are shown in Table 1.

Table 1. Comparison of results from runs A and B

Run	Q_p, W	T_w, °C	dR_f/dt, m^2 K/(W·day)	Nu ratio	Tumbler? Y/N
A	265	346	6.62E-05	1.5	N
B	264	347	6.15E-05	1.5	N
C	264	372	2.90E-04	1.2	Y
D	264	381	5.14E-04	1.1	Y

Nusselt Number Comparison

Noting the data in Table 1, when the tests reached steady state, the final wall temperature differed between the four runs despite having a consistent power input. The key impact of the varying wall temperatures was that the Nusselt numbers also varied. In Fig. 7, the Nusselt numbers obtained at t=0 from runs A through D are compared to the predicted values from the HTRI correlation.

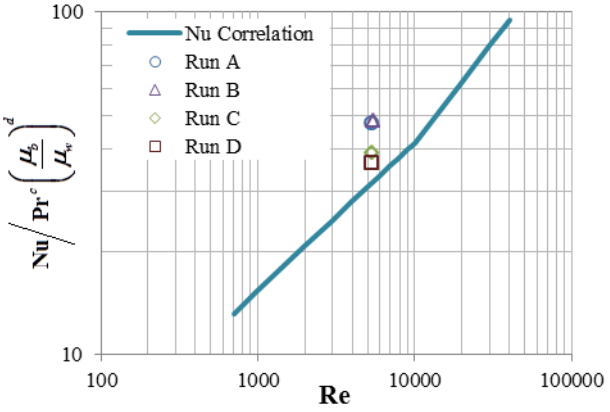

Fig. 7 Comparison of results from runs A through D to those of the Nusselt correlation

The differences in wall temperatures and the resulting Nusselt numbers between the runs with the fluid mixed with the rotary hand pump and those mixed with the tumbler suggest that the properties of the fluid were different between runs.

If the solids content of the fluid or stratification in the fluid was different due to poor mixing, then a difference in thermal conductivity, density, and/or heat capacity would be expected. The data suggest that the mixing methods did cause a difference in the homogeneity of the fluid.

It is possible that the crude oil from runs A and B contained more light components than the well-mixed crude oil from runs C and D. The lighter components could lead to boiling along the heated surface, resulting in a Nusselt number higher than expected. It is also possible that the increased particulate content of the well-mixed crude oil during these tests increases the ability for particulates to adhere to the heated probe before steady-state conditions are achieved. The fouling resistance would then increase, resulting in a lower Nusselt number at the start of steady-state conditions.

Fouling Rate and Deposition

In addition to comparing the Nusselt numbers from each run, the fouling curves were compared (Fig. 8).

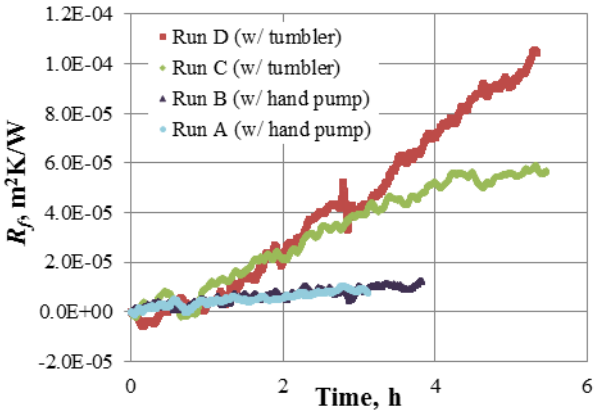

Fig. 8 Comparison of fouling curves from runs A through D

As seen in Fig. 8, runs C and D have noticeably higher fouling rates and higher final fouling resistances than runs A and B. These differences indicate that runs C and D had larger fouling deposits than runs A and B. It is possible that the higher fouling rates of runs C and D could have resulted from a higher concentration of solid particulates in the crude oil because they were better mixed than runs A and B. However, the change in fouling rate can likely be attributed to a 30 °C higher initial wall temperature in runs C and D compared to runs A and B. Runs C and D were above 370 °C, a temperature high enough for coking to occur (Scarborough et al., 1979), which would likely also increase the fouling rate. A visual comparison of the deposits on the probe is shown in Fig. 9.

Fig. 9 Comparison of fouling deposit from runs A and B

The images of the fouling deposits show that a heavier amount of fouling occurred during runs C and D than runs A and B. Qualitatively speaking, the deposits from runs C and D had a more granular texture, making them rougher than the deposits from runs A and B. The deposits from runs C and D are also visibly thicker than the fouling deposits collected during runs A and B. The differences in the deposits supports the fouling resistance data that were plotted in Fig. 8.

These differences in the extent of fouling between the tests runs with differently mixed crudes further confirm the importance of proper mixing. Even though the heat flux of the probe, the stirrer speed of the hollow cylinder, and the bulk temperature of the fluid were the same, the fouling observed was very different.

Repeatability

Based upon the fouling rate data, both mixing methods afforded good repeatability, as indicated in Figs. 8 and 9. The fouling rates and deposits for the crude oil mixed with the hand pump (A and B) were nearly identical. Likewise, the fouling rates for the crude mixed with the drum tumbler (C and D) were nearly identical for the first half of the tests, though they then changed three hours into the test. The reason for this change is unknown, but the similarity between the initial rates suggests repeatability. In addition, the fouling deposits for runs C and D have similar textures and suggest that both were in the coking regime.

CONCLUSIONS

Based on the experiments detailed in this report, we conclude the following:

1. Fouling rates, resistances, and deposits can differ greatly depending on the mixing method used.
2. Thorough mixing can change the particulate density of a crude oil.
3. Mixing crude oil with the drum tumbler produces repeatable results.
4. Variability of the Nusselt number between runs can be a sign of poor mixing. Attention should be given to the Nusselt number in future tests.
5. Visual inspection has shown that thorough mixing through rotation of a barrel greatly improves the mixing of a crude oil.

NOMENCLATURE

A	Heated area of probe, m^2
a	Constant, dimensionless
b	Constant, dimensionless
c	Constant, dimensionless
d	Constant, dimensionless
h	Heat transfer coefficient, $W/m^2 K$
k_f	Fluid thermal conductivity, W/m K
Nu	Nusselt number, dimensionless
Nu_{exp}	Experimental Nusselt number, dimensionless
Nu_{pred}	Predicted Nusselt number, dimensionless
P	Pressure, Pa
Pr	Prandtl number, dimensionless
Q_P	Probe power, W
R_f	Fouling resistance, m^2 K/W
Re	Reynolds number, dimensionless
t	Time, s
T_b	Bulk temperature, °C
T_w	Wall temperature, °C
μ	Fluid viscosity, Pa s
μ_b	Bulk fluid viscosity, Pa s
μ_w	Fluid viscosity at wall, Pa s
ω_o	Stirrer speed, rad/s

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