

## INVESTIGATING THE IMPACT OF BOILING CONDITIONS ON THE FOULING OF A CRUDE OIL

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### ABSTRACT

The impacts of nucleate boiling and pressure on crude oil fouling are factors that have not been heavily investigated in previous research. Variables such as wall temperature and fluid velocity/shear are often a main focus, as they are key variables for predictive fouling models, which provide insight to fouling thresholds.

Research detailed in this report shows that nucleate boiling and pressure greatly impact the measured fouling rate of a crude oil tested using HTRI's Rotating Fouling Unit (RFU). When nucleate boiling is occurring, the use of fouling resistance plots to measure fouling rates is not a reliable method due to the impact boiling has on the heat transfer coefficient. Visual inspection of fouling deposits to validate fouling resistance data has also been found to be critical. Images of fouling deposits are included. Fouling under non-boiling conditions was shown to increase with increasing pressure.

### INTRODUCTION

Crude oil fouling is an issue that is influenced by a wide variety of factors and variables, including the operating conditions of heat exchangers such as temperature, flow velocity, chemistry of the crude, and properties of the heat transfer surface. Whilst many variables have been explored in investigating crude oil fouling over the past 50+ years, little investigation into the effect of boiling on crude oil fouling has been conducted.

Boiling heat transfer is a complex topic, but when it occurs on a heat transfer surface with a growing fouling deposit that changes the surface roughness, analysis of fouling becomes particularly difficult. The predominant effect of nucleate boiling is that it raises the heat transfer coefficient. However, it is not fully understood if boiling conditions increase or decrease the fouling rate. It is possible that boiling could have an impact on the fouling rate in the following ways:

- Bubble formation increases turbulence at the surface that increases shear stress, thus reducing the rate of fouling through suppression or removal.
- The presence of gas bubbles due to boiling reduces the boundary layer resistance (transport limitations), making it easier for foulant particles or precursors to reach and adhere to the heat transfer surface.

### Previous Studies on the Effects of Boiling on Fouling

Asomaning (1997) observed subcooled boiling during fouling experiments conducted using a batch reactor system. In these experiments the heat transfer coefficient was found to increase with heat flux, resulting in reduced surface temperatures. However, no conclusion was made as to the impact of boiling on fouling. Fetisoff et al. (1982) and Crittenden and Khater (1984) observed cases with styrene and kerosene test fluids respectively where fouling was reduced under nucleate boiling conditions, compared to single-phase conditions. Ishiyama et al. (2013) carried out a thermo-hydraulic analysis on the effect of boiling on a crude oil heat exchanger also undergoing fouling, and found boiling conditions to cause the preheat train pump to reach its hydraulic limit faster than for single-phase flow conditions. This results in a reduced mass flowrate through an exchanger, potentially increasing fouling.

In 2012, HTRI collaborated with Bath University, and Harris (2014) was able to perform experiments using the Stirred Batch Cell (SBC). Compared to heavier crudes previously tested in the SBC, operating conditions for lighter crudes suggested that boiling was occurring; to achieve the same wall temperature, a higher power was required and a higher pressure was observed.

Also, it was found that flashing a certain proportion of the crude oil enabled the crude to begin fouling, where it would not before. Following flashing, a lower pressure rise during heating up and a lower power requirement to achieve target wall temperatures were also observed, suggesting a reduction in boiling.

### Boiling in Industrial Heat Exchangers

Whilst refineries typically operate crude oil distillation preheat train exchangers at pressures high enough to suppress boiling (20 – 27 bar), cases have been reported where the pressure was not sufficient to do so (Ishiyama et al., 2011; Zhang, 2013). It is likely that subcooled boiling often occurs on the surface of higher temperature heat exchanger tubes; however, these bubbles collapse in the bulk fluid itself. It is also possible that fouling in an exchanger could cause sufficient pressure drop during operation to reduce the pressure below the vapor pressure of lighter components in a crude oil, resulting in boiling. To remove the possibility of boiling, many refineries flash the crude oil in preflash drums shortly after the desalter

(Waintraub et al., 2007). However, not all crude distillation units have this capability; especially in the case of older refineries. Finally, the increasing use of ‘opportunity crudes’ such as shale oils in recent years, which often have large light fractions (Dion, 2014), further increases the possibility of boiling occurring during preheating.

### Nitrogen Pressurization Concerns

For over twenty years, HTRI has conducted fouling tests on the High Temperature Fouling Unit (HTFU). A substantial amount of data has been collected over the years, but minimal amounts of research have been performed on the impact of boiling on fouling.

The majority of fouling tests in the HTFU were run at elevated pressures of 34 bar and greater, using nitrogen to pressure the system (Longstaff et al., 2001) in order to suppress boiling. Recently however, this pressurization method has been questioned because concern was raised over the impact that added nitrogen may have on fouling data. The key concerns are:

- Dissolved nitrogen may boil on the fouling heat transfer surface. Fetissov et al. (1982) observed nitrogen boiling at the heat transfer with solutions of styrene in heptane.
- Dissolved nitrogen may impact the solubility of heavy oil components in the crude oil; this was previously suggested by Asomaning (1997).

However, if nitrogen is not used and tests are run at lower pressures, there is a concern about nucleate boiling along the heated surface. More research to determine the impact of pressurization and boiling on fouling is desired.

## METHODS

### The Rotating Fouling Unit (RFU)

From these cases, it was determined that further analysis on the impact of pressure on a fouling test was required. The RFU was chosen to evaluate the effect of pressure on the fouling curves of a crude oil in boiling and non-boiling conditions with minimal use of nitrogen to pressurize the system.

The RFU is a batch reactor fouling rig built based upon the unit used by Eaton and Lux (1984) and the SBC. A key difference between the units is that the RFU has a slightly larger capacity of 2.8 liters. Another key difference is that the hollow cylinder that creates fluid flow has a smaller gap between it and the heated metal probe than prior batch units. This means that it is capable of creating a higher shear stress at the heat transfer surface (a maximum of 3.2 Pa), making it more comparable to tubular fouling rigs and heat exchangers. The original design of the RFU is described in greater detail by Lane (2013). A detailed drawing of the unit can be seen in Fig. 2.

The RFU was designed with the intent of being a supplementary unit to the HTFU (an intube fouling rig) that could also be used to screen tests to be performed in the HTFU, albeit at lower shear stresses. Table 1 shows a comparison of the maximum operating conditions of the HTFU, SBC, and RFU.

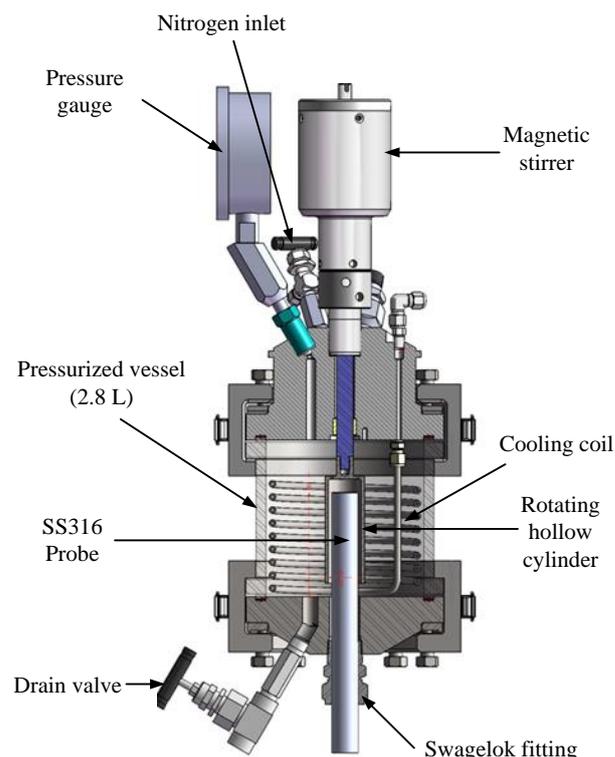


Fig. 1 A cross section of the RFU

Table 1. Maximum operating conditions of the HTFU, SBC, and RFU

|                         | HTFU | SBC | RFU |
|-------------------------|------|-----|-----|
| Bulk temperature, °C    | 300  | 343 | 280 |
| Surface temperature, °C | 500  | 400 | 482 |
| Pressure, bar           | 69   | 30  | 69  |
| Shear stress, Pa        | 15   | 1   | 3.3 |

In order to obtain repeatable and dependable results from the RFU, criteria-based protocols were created and used for all RFU tests. Before the oil drum was sampled, the drum was mixed on a drum tumbler for 15 minutes at ~20 RPM (Rotations Per Minute). The unit was started in two steps: bulk temperature and stir speed were established, and then heat was applied to the fouling probe. The unit was thoroughly washed with solvent between each test to ensure no carryover between runs. Determination of steady-state conditions and fouling rate followed methods described by Smith (2013).

### Pressurization of the RFU

The method by which the RFU is pressurized has been subject to scrutiny since the design phase commenced in 2012. As mentioned previously, HTRI has historically used nitrogen to pressurize fouling tests.

HTRI developed a pressurization method for the RFU that minimizes the use of nitrogen. Once the unit is completely emptied and dried out, a vacuum is pulled on the unit to provide the suction to draw crude oil into the unit. Then the crude oil is loaded into the RFU, and a small amount of nitrogen is added to break the vacuum and bring

the system to less than 0.7 bar. As the unit is heated, the crude expands, increasing the pressure. If the pressure is too high, then liquid can be removed from the system using the liquid sampling valve.

With this method, high pressures to suppress boiling can be achieved while the effects of added nitrogen are minimized. It is also possible to slightly under-fill the unit, so that the pressure does not rise as much when heated to promote boiling.

Regardless of the fill levels used in each experiment, it was verified that the crude oil covered both the probe and stirrer to promote recirculation of the fluid and ensure that the probe was always fully submerged in crude oil. Additionally, once the high temperature test conditions are reached, the thermal expansion of the fluid allows the fluid to fill the unit, minimizing the residual headspace. This means the amount of headspace for vapors to form in is negligible (< 2%).

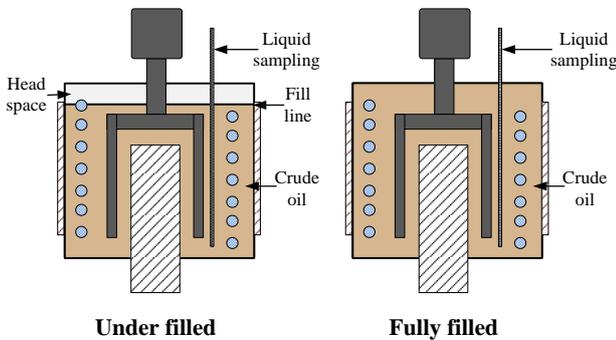


Fig. 2 Comparison of RFU fill level

### Determination of Boiling using the Nusselt number

The primary way to denote whether boiling occurred during an experiment in the RFU is to look at the Nusselt number. When the RFU was first built, shakedown tests were performed with p-xylene and Duratherm to determine the dimensionless heat transfer coefficient correlation as a function of Reynolds number for single-phase heat transfer. The RFU was treated as a Taylor-Couette system, and the Reynolds number was calculated using the physical properties of the crude oil provided by the crude oil donor as follows:

$$Re = \frac{\rho(R_o - R_i)R_o\omega_o}{\mu} \quad (1)$$

After the Nusselt number was plotted as a function of the Reynolds number, a correlation was developed specifically for the RFU. This correlation is used to predict the Nusselt number in the RFU at various Reynolds numbers. To show the trend with the Reynolds number, the Nusselt number is divided by the Prandtl number and viscosity ratio terms. The results from these shakedown tests are shown in Fig. 3, and the Nusselt number correlation is fit to the data using

$$Nu = a Re^b \left( Pr^c \left( \frac{\mu_b}{\mu_w} \right)^d \right) \quad (2)$$

where  $a$ ,  $b$ ,  $c$ , and  $d$  are dimensionless constants.

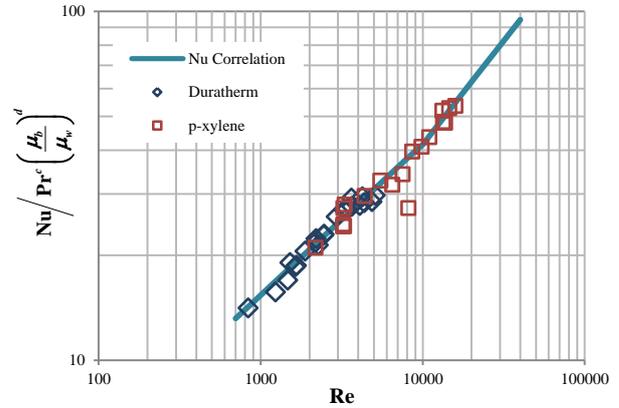


Fig. 3 Heat transfer coefficient correlation from shakedown testing of RFU with p-xylene and Duratherm

If two-phase heat transfer occurs, the Nusselt number when plotted on the above graph should be greater than the correlation. Comparing the ratio of the measured Nusselt number to that predicted by the correlation at a given Reynolds number gives a quantitative indication of the extent of boiling.

### EXPERIMENTAL SUMMARY

The aim of the tests conducted in the RFU was to better understand how pressure and boiling conditions affect fouling deposition of a single crude oil. The properties of the crude oil tested are found below in Table 2.

Table 2. Physical properties of crude oil used in experiments

| Property             | Value     | Method           |
|----------------------|-----------|------------------|
| API at 15.6°C        | 37.6 °    | ASTM D-287/5002  |
| Specific gravity     | 0.8367    | ASTM D-1298/5002 |
| Viscosity at 26.7 °C | 4.17 cSt  | ASTM D-445       |
| Viscosity at 50 °C   | 2.63 cSt  | ASTM D-445       |
| Saturates            | 54.85 WT% | ASTM D-4124      |
| Aromatics            | 37.52 WT% | ASTM D-4124      |
| Resins               | 6.65 WT%  | ASTM D-4124      |
| Asphaltenes          | 0.98 WT%  | ASTM D-4124      |
| Total sulfur         | 0.964 WT% | ASTM D-4294      |

Additionally, the crude oil was found to be self-incompatible using the Wiehe incompatibility index.

To determine the impact of pressurization on a fouling test, multiple tests were run in the RFU at various pressures while a constant bulk temperature and stirring speed were maintained. The wall temperature was intended to be the same for each run. However, at lower pressures, a high wattage was required to obtain the same wall temperature as the higher pressure tests (D – F). Two tests (A and B) were

performed at a similar wall temperature (~304 °C), while run C was performed at a similar probe power (160 W) compared to the higher pressure tests (D – F). The extent of boiling can be compared between these runs. The conditions run are shown in Table 3.

Table 3. Operating conditions of each test performed in the RFU

| Run | $Q_p, W$ | Stirring speed, RPM | $T_b, °C$ | $T_w, °C$ | $T_{bp}, °C$ | $P, bar$ |
|-----|----------|---------------------|-----------|-----------|--------------|----------|
| A   | 490      | 500                 | 226       | 304       | 266          | 10.3     |
| B   | 490      | 500                 | 226       | 294       | 287          | 14.5     |
| C   | 160      | 500                 | 226       | 263       | 282          | 13.2     |
| D   | 160      | 500                 | 226       | 306       | 366          | 28.0     |
| E   | 160      | 500                 | 226       | 306       | 432          | 41.4     |
| F   | 160      | 500                 | 226       | 308       | 478          | 57.2     |

**RESULTS AND DISCUSSION**

For all the tests carried out, the Nu ratio and fouling data are presented in Table 4.

Table 4. Calculated Nusselt ratios, fouling rates, fouling resistance uncertainties, and final fouling resistances obtained from RFU tests

| Run | Nu ratio | $dR_f/dt$<br>m <sup>2</sup> K/(W day) | $dR_f/dt$<br>uncertainty | Final $R_f$ ,<br>m <sup>2</sup> K/W |
|-----|----------|---------------------------------------|--------------------------|-------------------------------------|
| A   | 4.2      | 1.62E-04                              | 2.16E-07                 | 3E-05                               |
| B   | 4.58     | 1.85E-04                              | 2.97E-07                 | 3.95E-05                            |
| C   | 2.83     | 2.78E-04                              | 7.66E-07                 | 6.2E-05                             |
| D   | 1.4      | 0.19E-04                              | 2.90E-07                 | 0.22E-05                            |
| E   | 1.39     | 0.77 E-04                             | 14.20E-07                | 0.18E-05                            |
| F   | 1.30     | 2.05E-04                              | 4.50E-07                 | 4.53E-05                            |

**Determination of Boiling in the RFU**

A Nusselt ratio significantly greater than 1 (i.e., > 1.5) suggests boiling, while a Nusselt ratio closer to 1 (< 1.25) indicates single-phase heat transfer. However, for Nusselt ratios between 1.25 – 1.5 it is less certain whether boiling is present. The high pressure runs, D – F, all had Nu ratios between 1.3 and 1.4 over a 29.2 bar range. Based on the Nu ratio alone, the phase state of runs D – F is uncertain.

By comparison, the lower pressure runs, A – C, had higher Nusselt ratios, suggesting two-phase heat transfer. runs D – F had a greater variation in the ratios (2.83 – 4.58). Even though these runs were at similar pressures, run C had a noticeably lower Nusselt number than runs A and B because of its lower wall temperature (263 °C), which likely lessened the extent of boiling. These results are also plotted in Fig. 4, compared to Eqn. (2).

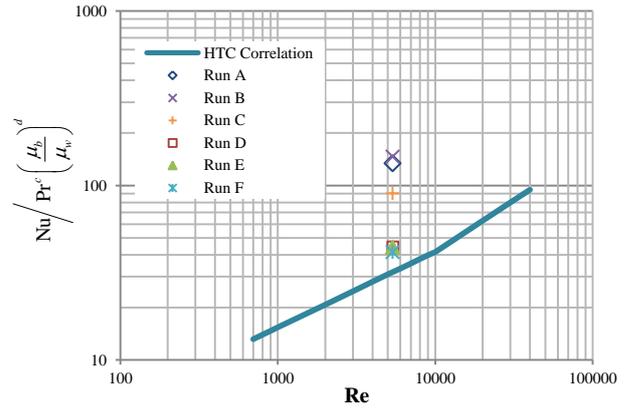


Fig. 4 Comparison of heat transfer coefficient from crude oil runs in RFU to the heat transfer coefficient correlation

To support these observations, the crude oil bubble point curve was predicted using the property simulation package, VMGThermo™, based upon distillation data for the crude at ambient pressure measured. These data are plotted in Fig. 5. The line represents the transition from single-phase to two-phase conditions.

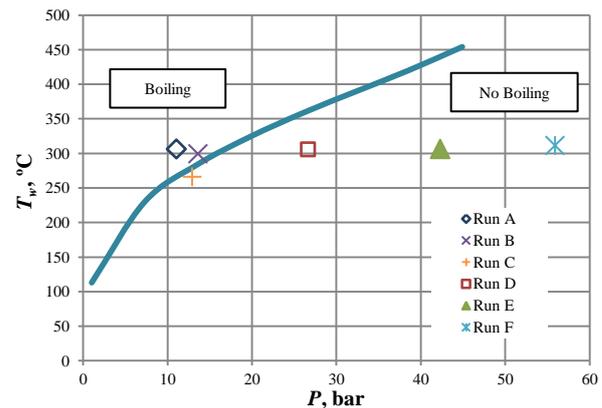


Fig. 5 Comparison of conditions for each run on the RFU to the simulated bubble point curve of the crude tested

Data points for runs A – C, where boiling is thought to have occurred, are in the boiling region or very close to the bubble point. The higher pressure data points for runs D – F are well into the single-phase area of the plot. The graph supports the findings of the experiments.

It should be noted that because this graph is based upon a simulation extrapolating from measured ambient pressure data points, the bubble point line has a degree of error in it. However, data points for runs D – F, whose phase states were uncertain, are well into the single-phase region of the graph. Thus, we conclude these runs did not have boiling. While the other data points for runs A – C are closer to the bubble point curve, their Nusselt ratios are high enough (> 1.5) to confirm that boiling was occurring even though run C lies slightly within the bubble point curve.

**Impact of Pressure on Fouling Curves at Non-boiling Conditions**

For single-phase flow, high pressure tests (D – F), linear fouling trends were observed and the fouling rate was determined to be affected by pressure. The fouling plots for these three runs are shown in Fig. 6.

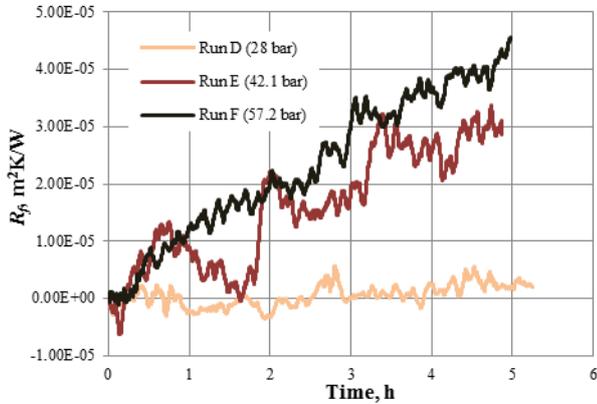


Fig. 6 Fouling curves for the three runs without boiling

Significant fouling deposits were observed on the heat transfer surface suggesting that the measured fouling resistances were genuine. Images of the deposition area for runs D – F are shown in Fig. 7. Currently, there is no method at HTRI to quantitatively analyze the deposits.



Fig. 7 Comparison of deposits obtained from test runs without boiling

The time zero point of the fouling data shown in the graphs was determined using the methods detailed in Smith (2013). The time zero point is that at which the test conditions become stable-this includes the bulk temperature, stirrer speed, heater power and pressure. Fouling rates were calculated as a linear average from the start to the end of the test. The calculated linear fouling rates are plotted as a function of pressure in Fig. 8.

It is noted that Run E had a greater amount of inflections than the other runs; it is not known what the cause of this was. Despite the occasional downturns in  $R_f$  for run E, there was still a distinct overall linear trend in fouling rate over the duration of this test, especially in the final 4 hours.

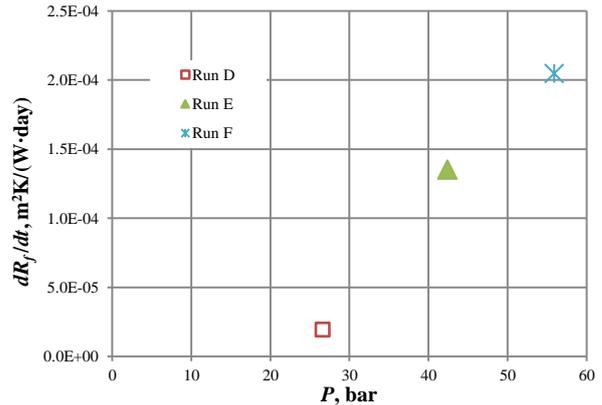


Fig. 8 Fouling rate vs. pressure for runs D – F

Fig. 8 indicates that the fouling rate of a crude oil increases as the pressure increases and suggests that the fouling rate could be predicted as a function of pressure under single-phase heat transfer conditions. This is similar to what was observed by Crittenden and Khater (1984) in their fouling experiments with kerosene. This trend could be explained by the impact of pressure on asphaltene precipitation as previously suggested by Burke et al. (1990) and De Boer et al. (1995). It is feasible that the elevated pressures caused asphaltenes to precipitate, increasing the fouling rate. This finding suggests that if fouling experiments are carried out at an elevated pressure (higher than typically found in refinery heat exchangers), then fouling rates at these pressures would be expected to be higher.

**Impact of Boiling on Fouling Curves**

For the data points where boiling was occurring (A – C), high positive fouling rates were observed. These are plotted in Fig. 9.

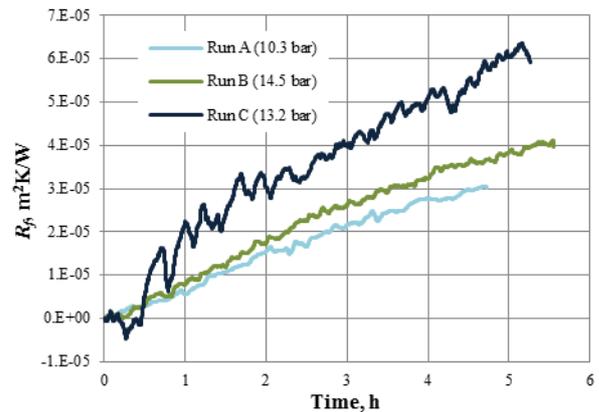


Fig. 9 Fouling curves for the three runs experiencing boiling

Whilst these fouling trends suggest a high fouling rate for the conditions, little to no fouling deposit was observed, especially compared to later tests where complete coverage of the heated surface was observed. Images of the deposition area for runs A – C are shown in Fig. 10.

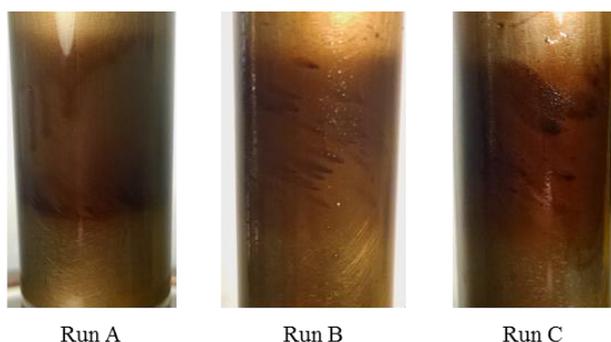


Fig. 10 Comparison of deposits obtained from test runs with boiling

Other variables such as the probe power and bulk temperature were well controlled, suggesting that the positive fouling curves were not caused by unstable operation.

#### **False fouling curves**

In Fig. 9, positive fouling rates are noticeable for runs A – C, similar to the rate observed in run F. However, the images in Fig. 10 show only staining from the crude oil along the heated section, with the metal surface still visible, as opposed to the more distinct deposits in Fig. 7. Notably, runs B and F have nominally the same end fouling resistance; thus, it would be expected that both have the same amount of deposit. However, very little deposit accumulated along the surface of the probe during runs A – C, suggesting that the positive fouling rates in Fig. 9 did not accurately represent the amount of deposit seen on the heated surface. The occurrence of boiling seems to have decreased the convective heat transfer coefficient throughout the course of the experiment and caused a false fouling curve.

Some small agglomerations of particulates were deposited on the heat transfer surface, but the surface coverage was incomplete, suggesting that fouling was still in the induction phase. Typically, the presence of small particulates on the heat transfer surface increases turbulence at the heated surface due to surface roughening, which should then reduce the measured fouling resistance due to a resultant increase in heat transfer coefficient. Also, these small particulates would be expected to act as nucleation points for the bubbles under boiling conditions, which would increase the extent of boiling and the heat transfer coefficient, further reducing the fouling resistance. The boiling heat transfer coefficient is a strong function of a temperature difference between heat transfer surface and bulk fluid. Consequently, small amount of deposit on heat transfer surface will have significant impact on the boiling heat transfer coefficient.

It is possible that the small agglomerations of fouling particulates created a smoother surface compared to the underlying metal surface, thereby reducing the heat transfer coefficient and the number of nucleation sites, resulting in an increasing measured fouling resistance. There was no way of testing this hypothesis, however.

Because the convective heat transfer mechanism is different between single-phase and two-phase heat transfer, thermally analyzing fouling rates based upon a change in the heat transfer coefficient is clearly difficult due to the large impact boiling has on the heat transfer coefficient.

#### **CONCLUSIONS AND RECOMMENDATIONS**

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

1. A method was developed to pressurize HTRI's RFU to pressures up to 69 bar with minimal usage of nitrogen gas by using the thermal expansion of a test fluid in a full vessel.
2. If boiling is allowed to occur during fouling experiments, fouling resistance plots based upon change in the heat transfer coefficient may be an unreliable measure of the deposit's thermal resistance.
3. Physical inspection of fouling deposits is important in order to validate fouling resistance data in case a false fouling resistance is recorded.
4. Pressure can affect the fouling rate, with higher fouling rates observed at higher pressures; it is possible this was caused by decreased asphaltene solubility at higher pressures.
5. For studies unrelated to boiling/pressure, it is important to keep pressure consistent between fouling tests in order to keep data comparable and maintain a stable pressure throughout the course of the test.
6. Little to no fouling (only a few small agglomerations of deposited particles) was observed on the heat transfer surface when nucleate boiling was occurring, compared to complete coverage for single-phase conditions.
7. Because fouling behavior is different when nucleate boiling occurs, knowing when nucleate boiling is occurring in a test rig or exchanger is critical.
8. Pressure and its impact on asphaltene solubility and nucleate boiling is an important variable to consider in the development of future fouling models.
9. Additional testing needs to be performed at a wide variety of bulk temperatures, wall temperatures, and shear stresses to further investigate the relationship between pressure and fouling rate.

#### **NOMENCLATURE**

|       |                               |
|-------|-------------------------------|
| $a$   | Constant, dimensionless       |
| $b$   | Constant, dimensionless       |
| $c$   | Constant, dimensionless       |
| $d$   | Constant, dimensionless       |
| Nu    | Nusselt number, dimensionless |
| $P$   | Pressure, bar                 |
| Pr    | Prandtl number, dimensionless |
| $Q_p$ | Probe power, W                |
| $R_f$ | Fouling resistance, $m^2 K/W$ |

|            |                                  |
|------------|----------------------------------|
| $R_i$      | Inner radius, m                  |
| $R_o$      | Outer radius, m                  |
| Re         | Reynolds number, dimensionless   |
| $t$        | Time, s                          |
| $T_b$      | Bulk temperature, °C             |
| $T_{bp}$   | Boiling point temperature, °C    |
| $T_w$      | Wall temperature, °C             |
| $\mu$      | Fluid viscosity, Pa s            |
| $\mu_b$    | Bulk fluid viscosity, Pa s       |
| $\mu_w$    | Fluid viscosity at wall, Pa s    |
| $\rho$     | Fluid density, kg/m <sup>3</sup> |
| $\omega_o$ | Stirrer speed, rad/s             |

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