ABSTRACT
A 2019 study was the first of its kind investigating how a benchtop unit’s mode of operation impacts a variety of fouling parameters related to crude oil fouling. Running the test in the recirculating mode was hypothesized to induce two competing mechanisms: thermal stress on the sample which could contribute to fouling, and depletion of precursor which could reduce fouling compared to the once-through mode. To achieve the once-through versus recirculating comparison while holding time as constant, different flow rates were used. Subsequently, the current study aims to confirm the previous conclusions while holding the flowrate constant and increasing the number of cycles that the crude will flow through the unit. The study found that at constant flowrate the same conclusion can be made: recirculating the feed affects the deposit characteristics and fouling behavior.

INTRODUCTION
The 2019 study compared the results of fouling experiments performed in both the recirculating and once-through modes of operation. The variables observed were fluid properties before and after testing, analysis of the deposits retrieved from the test tubes, and fouling resistance complemented by visual observation of fouling deposits. Running the test in the recirculating mode was hypothesized to induce two competing mechanisms: thermal stress on the sample which could contribute to fouling, and depletion of precursor which could reduce fouling compared to the once-through mode. Experimental results indicated that thermal stress induced from running the test in the recirculating mode took precedent over the depletion of fouling precursor. This conclusion was supported by the results from the deposit analysis and fluid characterization.

However, in this current study, we aim to investigate the differences, if any, in fouling behavior and deposit composition when running tests keeping flowrate constant. In the previous study, time was held constant, but here we will extend time of the run in the recirculating tests to ensure a minimum of 4 cycles of feed are recirculated.

EXPERIMENTAL SECTION
Fouling Test
The fouling experiments were performed in the same test rig as the 2019 test runs (Cibotti, 2019). The test rig utilizes an electrically heated vertical tube shaped coupon equipped with multiple stationary thermocouples measuring surface temperatures. The test coupon is inserted into an outer tube shaped test section with process fluid flowing upward in the annulus. A new coupon is used for each experiment and deposit weights are determined by weight measurements of the test sections before and after the experiments (Cibotti, 2019). The overall heat transfer coefficients $U_{(t,0)}$ are calculated based on heat balance where mean temperature difference is obtained from fluid inlet, fluid outlet, and heat transfer surface temperature measurements. As done previously, the fouling experiments were performed at a constant surface temperature. Both modes utilize the same startup procedures and ramp up times for arriving at the control temperature.

In the recirculating experiments, the fluid sample passes several times through the test section, imposing thermal cycling that impacts P-value and subsequent fouling behavior.

The once-through experiments are designed to behave similarly to the refinery preheat train exchangers where fresh feed is constantly introduced (Jackowski, 2017).

The previous experiments were performed at constant surface temperature, pressure, and duration but varied in flowrate and sample volume to do so. Keeping the test duration constant for both modes of operation allowed for a proper comparison of collected deposit samples and deposit weights. However, the experiment design failed to consider the effects, if any, of the change in flowrate had on fouling. Thus, the continuation of this study focused on holding flowrate constant and examining the effects of recirculation on the compatibility and stability of the blend.

<table>
<thead>
<tr>
<th>Test Rig Operating Conditions</th>
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</thead>
<tbody>
<tr>
<td>Velocity (mm/s)</td>
</tr>
<tr>
<td>Reynolds number at average bulk temp. (ø)</td>
</tr>
<tr>
<td>Bulk Inlet Temperature (°C)</td>
</tr>
<tr>
<td>Maximum Surface Temperature (°C)</td>
</tr>
<tr>
<td>Heat Flux (kW/m²)</td>
</tr>
<tr>
<td>Operating Pressure, MPa</td>
</tr>
<tr>
<td>Shear Stress, Pa</td>
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</tbody>
</table>

<table>
<thead>
<tr>
<th>Figure 1. Test Rig Operating Conditions</th>
</tr>
</thead>
<tbody>
<tr>
<td>Study</td>
</tr>
<tr>
<td>-------</td>
</tr>
<tr>
<td>2019</td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td>2022</td>
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Figure 2. Test Duration, Flow Rates, and Cycles
Fluids Characterization

Crude oils were characterized using the same conventional techniques as in 2019 (Rogel, 2003). A list of characteristics and how they were measured are shown in Figure 3. Additionally, compatibility parameters were determined for crude oils before and after the fouling testing.

![Figure 3. Characterization of crude oil](image)

<table>
<thead>
<tr>
<th>Properties</th>
<th>ASTM Method</th>
<th>D4052</th>
<th>D5762</th>
<th>D1552</th>
<th>D6560</th>
</tr>
</thead>
<tbody>
<tr>
<td>API gravity</td>
<td>32</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Nitrogen (ppm)</td>
<td>1900</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sulfur (wt. %)</td>
<td>1.6</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Asphaltene Content (wt. %)</td>
<td>3.2</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

As discussed in the last paper and seen in the previous study, it is understood that composition plays a significant role in the colloidal stability of petroleum systems (Pfeiffer, 1940). The characteristics of the asphaltenes and the solvent power of the oil are considered fundamental factors for the stabilization of asphaltenes in crude oil (Hatke et al. 1993).

\[ FR = \frac{V_t}{V_t + V_h} \]  
\[ C = \frac{W_a}{V_t + V_h} \]

In a typical test, values of FR (equation 1) are plotted as a function of the concentration (equation 2). In this plot, the point at which the line intercepts the x-axis is defined as \( C_{\text{min}} \), and the y-intercept is \( FR_{\text{max}} \). \( C_{\text{min}} \) represents the onset of the precipitation when the crude oil is titrated without dilution. This value is frequently determined by extrapolation since an experimental value might not be available due to setup limitations (Heithaus, 1962).

The Heithaus compatibility parameters are calculated as:

\[ P_a = 1 - FR_{\text{max}} \]  
\[ P_0 = FR_{\text{max}}[(1/C_{\text{min}})+1] \]  
\[ P = P_0 / (1 - P_a) \]

Note that \( P > 1 \) means the system is stable.

RESULTS AND DISCUSSION

The objective of this research was a continuation of a previous study that compared the fouling behaviors of a crude batch tested in two modes of operation, one in which the crude ran once-through the test rig, and the other which had the crude recirculating throughout the duration of the test. This subsequent study looked at the effects of keeping flowrate constant rather than duration constant. The analysis embodied a holistic approach to fouling testing which took into account visual fouling observations and fluid characterization in addition to heat transfer data. Looking at the fouling resistance curves alone does not yield a firm conclusion on the effects of switching from a recirculating to a once-through mode of operation. However, the characteristics of the deposited material on the tube indicate that recirculating the crude sample results in more insoluble deposition. This can be observed visually in Figure 8 which compares the morphology of the deposits from the recirculating versus the once-through tests. The recirculating tests’ deposits appear to have more solid material deposition that adheres to the tube compared to the once-through test. The recirculating tests have noticeably more insoluble deposits. This conclusion is also supported by the theory initially stated which suggested that thermal stress (the cyclical heating of the fluid in the recirculating tests) affects the stability of the crude which may cause material to drop...
Fouling resistance plots for each run are plotted in Figures 6 and 7. Equation (6) calculates fouling resistance, \( R_f \), by:

\[
R_f(t) = \frac{1}{U_f(t)} - \frac{1}{U_0}
\]  

(6)

![Figure 6. Fouling Resistance plots of 2022 study.](image)

![Figure 7. Fouling Resistance plots of 2019 study.](image)

Figures 6 and 7 show the fouling resistance plots of all experiments. All tests begin with relatively similar fouling behavior until the 5 hour mark. Observing the fouling resistance curve for the recirculating test, after 5 hours the fouling resistance drops off which may signify removal of deposit. After the dip the fouling resistance increases at a lower rate but exhibits more linear behavior. The once-through test has a more steady trend in fouling resistance compared to the recirculating run which was expected and in agreement with the previous results. The recirculating curve is initially steeper, speculated to be caused by foulant in the feed being deposited early on before the feed cycles through several times. Note that for the once-through test, the maximum duration at 2.5mL/min was roughly 10 hours.

In addition to analyzing heat transfer data, visual inspection of the tubes was performed. Figure 8 shows images of the tubes after each test concluded. The test section was carefully removed from the test rig, the tubes removed, and the images were taken on a digital microscope. Visually there is a difference in the morphology of between the deposit left from the recirculating tests versus once-through test. This corroborates with the conclusion of the previous study where the once-through test produced more soluble deposit and less insoluble deposit than the recirculating tests.

![Figure 8. Images of tubes extracted from the test section after conclusion of each test in 2022](image)

**CONCLUSION**

This study was a continuation of a novel 2019 study investigating the impact the mode of operation has on fouling. Running tests in the recirculating mode was found to induce thermal stress on the sample which contributed to more insoluble material recovered from the tube.

Peer review and discussion of findings from the 2019 study led to the investigation performed this year. The question of ‘how does holding flow rate constant impact these findings’ was asked at the 2019 Heat Exchanger Fouling and Cleaning Conference and in the subsequent peer review. The hypothesis was that because the test is run at such low flowrate, negligible shear stress can be assumed. Thus, increasing flow rate should also have negligible effects. The current study corroborates with this hypothesis given that changes in compatibility parameters and fouling resistance curves demonstrated to be very similar compared to the variable flowrate study in 2019.

Future testing in this realm will investigate the effects of ageing unstable crude samples to bring awareness to reactivity at ambient conditions.

**NOMENCLATURE**

- FR: Flocculation ratio
- \( FR_{\text{max}} \): Y-intercept of the Flocculation Ratio
- \( V_t \): Volume of toluene, mL
- \( V_h \): Volume of heptane, mL
- \( C \): Concentration of solution, g/mL
- \( C_{\text{min}} \): The onset of precipitation when the crude oil is titrated without dilution
Wa  Mass of crude oil, g
Pa  Peptizability of asphaltenes
Po  Solvent power of maltenes
P   Overall compatibility of the system
Rf  Fouling resistance, m²K/W

U(t)  Overall heat transfer coefficient at time t, W/m²-K
U(0)  Overall heat transfer coefficient at time 0, W/m²-K

REFERENCES


