ANNULAR HEATING PROBES IN OIL FOULING: EFFECTS OF WALL SHEAR STRESS

by

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Abstract

Fouling, as the deposition of undesired material on a surface, is a common reason for energy loss in oil refineries, particularly in the preheat train section, where the surface of heat exchangers become fouled. Some of the major issues associated with fouling are the increase in pressure drop and thermal resistance.

Fouling rates are known to be a function of oil properties, temperature, and fluid flow properties. There is not yet an agreement among researchers on the determining flow property (Reynolds number, velocity, and inner wall shear stress) of the oil fouling process. The present work focuses on annular flow, which is commonly used for fouling studies, and discusses the effect of inner wall shear stress on fouling rate. An experimental study was undertaken in which an asphaltene containing oil blend (ATB) was recirculated in a flow loop equipped with an annular heat transfer test section. The change in overall heat transfer coefficient was monitored at different flow conditions to calculate the fouling resistance and rate.

First, a series of asphaltene precipitation tests was carried out to identify a preferred diluent to be used with the highly viscous ATB.

The existing flow loop was modified to accommodate a new pump, and a new data acquisition system was installed. A new 82 cm long annular fouling probe system was devised based on a commercially available 11 cm long 1000 W electrical heater equipped with a central core thermocouple. With Probe #1, fouling was not detected in a series of runs with power up to 412 W. Surface temperatures were determined to be low by Wilson-plot calibration and heat conduction calculations. Probe #2, with an improved design, showed lower internal thermal resistance, and hence higher surface temperature at a given power. As fouling was detected in one experiment, this probe design appears promising for future fouling studies.
The literature on inner wall shear stress calculation for concentric annuli was reviewed, and appropriate equations selected. Inner wall shear stress was calculated for four various fouling studies carried out at UBC. Fouling rate dependencies on velocity, Reynolds number, and wall shear stress were compared.
Preface

The present thesis was developed by the author under supervision of Dr. A. Paul Watkinson. The author did the literature review, modified the experimental setup, performed experiments, and analyzed the data to prepare the thesis in Chemical and Biological Engineering Department of the University of British Columbia.
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<td>Radius</td>
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<td>$U_{fouled}$</td>
<td>Overall heat transfer coefficient in fouled condition</td>
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<tr>
<td>$v$</td>
<td>Velocity</td>
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<tr>
<td>$v_{av}$</td>
<td>Average velocity</td>
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<tr>
<td>$v_{bulk}$</td>
<td>Fluid bulk velocity</td>
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<tr>
<td>$Vol$</td>
<td>Volume</td>
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<tr>
<td>$x$</td>
<td>Deposit thickness</td>
</tr>
<tr>
<td>$\rho$</td>
<td>Density</td>
</tr>
<tr>
<td>$\rho_f$</td>
<td>Fouling density</td>
</tr>
<tr>
<td>$\tau_{in}, \tau_{out}$</td>
<td>Inner, outer wall shear stress</td>
</tr>
</tbody>
</table>
\( \tau_w \)  
Wall shear stress, (Pa)

\( \varphi^* \)  
Geometry function

\( \mu \)  
Viscosity, (kg/m.s)

\( \mu_w \)  
Viscosity at wall temperature, (kg/m.s)

\( \delta \)  
Solubility parameter, (MPa)\(^{0.5}\)

\( \delta_{cr} \)  
Critical solubility parameter, (MPa)\(^{0.5}\)

\( \delta_f \)  
Flocculation solubility parameter, (MPa)\(^{0.5}\)

\( \delta_H, \delta_T \)  
Solubility parameter of n-Heptane, Toluene, (MPa)\(^{0.5}\)

\( \delta_o, \delta_p, \delta_s \)  
Solubility parameter of oil, precipitant, solvent, (MPa)\(^{0.5}\)

\( \lambda \)  
Dimensionless radial distance of maximum velocity in annulus, \( r_{\text{max}}/r_{\text{out}} \)

\( \nu \)  
Kinematic viscosity, (cSt)

\( \varphi \)  
Volume fraction

\( \varphi_o, \varphi_p, \varphi_s \)  
Volume fraction of oil, precipitant, solvent

\( \Delta T_m \)  
Mean temperature difference, (°C)

\( \Delta T_{m,\text{clean}} \)  
Mean temperature difference in clean condition, (°C)

\( \Delta T_{m,\text{fouled}} \)  
Mean temperature difference in fouled condition, (°C)

\( \varepsilon \)  
Radius ratio, \( r/r_{\text{out}} \)
List of Abbreviations

ATB    Atmospheric Tower Bottoms
CII    Colloidal Instability Index
HFD    Hydro-dynamically Fully Developed
LSB    Light Sour Blend oil
TFD    Thermally Fully Developed
PFX    Paraflex
Acknowledgements

First and foremost, I offer my gratitude to my supervisor Dr. A. Paul Watkinson. Without his supervision, guidance, and support I would not have been able to accomplish this research project. It was a great honor for me to work with him.

My special thanks goes to Dr. Yonghua Li who has not only guided me through the experiments but has been a great mentor to me since I started working.

I would also like to thank my colleague, Wei Wang, for sharing all his experience and knowledge with me, as well as laboratory equipment!

Financial support provided by NSERC is gratefully acknowledged.

Finally, I wish to gratefully thank my husband, for his endless love and support throughout my professional life. This work would not have been possible without him.
Dedication

_I dedicate this thesis to my dearest, Hamidreza, for his priceless support._
Chapter 1: Introduction

Fouling, the accumulation of unwanted solid material on a surface, is of great importance in the oil industry due to the decreased efficiency associated with it. Energy conservation is an important factor in the economics of industrial processes and to compensate the efficiency loss due to the fouling, more energy is used. Refiners could face fouling at any step in petroleum processing. The problem of fouling represents a challenge to designers and process engineers in terms of heat transfer technology, economics and environmental acceptability. 1.1 shows some examples of heat exchanger fouling.

(a) Right side-Before fouling, Left side- After fouling

(b) Fouling inside a heat exchanger

1.1 Some examples of the heat exchangers with fouling
1.1.1 Fouling cost in refineries

In the refining of crude oil, 6% of the energy content of each crude oil barrel is used for its refinery (Macchietto et al., 2011). For a global production of about 82-85 million barrels per day (bbl/day), an amount of energy equal to the entire production of Exxon or Shell is needed to operate 720 refineries all around the world. Figure 1.2 shows the approximate annual fouling cost in some countries (Tay and Yang, 2006). Fouling results in extra expenditures on different levels that will be discussed later in this work. Any reduction in these costs makes the refining process less expensive.

![Figure 1.2 Annual cost of fouling in some countries (1992 estimation)](image)

Crude oil distillation unit (CDU), the first processing unit in all refineries, consumes a large amount of this energy. A network of heat exchangers, the preheat train (shown in Figure 1.3), which heats the crude oil to the temperature required for the atmospheric distillation
unit, uses the heat of the product and internal streams of the distillation column, to recover a part of the energy that is consumed in this unit. The preheat train has a great potential for fouling because of the impurities in crude oil which tend to deposit as fouling layers on the tube surfaces.

**Figure 1.3** A typical preheat train network in refinery with seven heat exchangers. The heat is provided by products that need to be cooled and internal streams.

The growth of fouling layers over time decreases the energy recovery in the pre-heat train, resulting in an increasing energy demand in the furnace in order to meet the set-point temperature of the oil which enters the distillation column. Assuming that just one part per million of the impurities in the crude oil deposits on the surface of these heat exchangers, for a refinery with 235,000 barrels processing per day, having 140 kg oil in each barrel, gives rise to 32 kg of deposits daily. At the end of a year, 11 tonnes of deposits would have accumulated if the refinery runs 350 days a year. The magnitude of this number indicates that removing and disposing of deposits from pre-heat trains is not a simple matter.
Generally the cost related to fouling can be summarized as follows:

1. Higher capital investment due to the excess surface area needed to accommodate fouling, costs of transportation and installation of bigger plants.

   Larger heat transfer surface is needed to have the same amount of heat transfer when fouling occurs in the system. The following equations show the relation between clean condition area and fouled condition area.

\[
 Q = A_{\text{clean}} U_{\text{clean}} \Delta T_m = A_{\text{fouled}} U_{\text{fouled}} \Delta T_m \quad \text{for constant } \Delta T_m \quad (1.1)
\]

Therefore,

\[
 \frac{A_{\text{fouled}}}{A_{\text{clean}}} = \frac{U_{\text{clean}}}{U_{\text{fouled}}} \quad (1.2)
\]

Considering that fouling rate is represented by

\[
 R_{\text{fouling}} = \frac{1}{U_{\text{fouled}}} - \frac{1}{U_{\text{clean}}} \quad (1.3)
\]

Hence,

\[
 \frac{A_{\text{fouled}}}{A_{\text{cleaned}}} = 1 + R_{\text{fouling}} U_{\text{clean}} \quad (1.4)
\]

To have same amount of heat transfer when fouling exists, the tubular exchangers are typically designed 20-50% oversized. This value for compact heat exchangers is 15-20% (Awad, 2011). In this approach to accommodate fouling, the overall heat transfer coefficient is determined for clean conditions, and the surface area required for clean condition is increased by a percentage.
Moreover in cases where excessive fouling is expected it might be necessary to install a standby heat exchanger, so that one heat exchanger can be in service while the other is being cleaned.

2. Energy losses as a result of decrease in thermal efficiency and increase in the pressure drop.

As mentioned in the introduction, presence of the fouling material decreases the heat transfer ability of the unit. Additional energy would be required to compensate the efficiency loss, as is also evident from equation 1.6 once a heat exchanger is designed with a given area $A$, if the heat transfer rate is to be maintained ($Q_{fouled} = Q_{clean}$), in the presence of fouling (decreasing $U$) the driving force for heat transfer ($\Delta T_m$) must be increased.

\[
\frac{Q_{fouled}}{Q_{clean}} = \frac{U_{fouled}\Delta T_{m,fouled}}{U_{clean}\Delta T_{m,clean}} = 1
\]  
\[1.5\]

\[
\frac{\Delta T_{m,fouled}}{\Delta T_{m,clean}} = \frac{U_{clean}}{U_{fouled}} \geq 1
\]  
\[1.6\]

Besides, the presence of deposit on the surface increases the pressure drop, as a result additional pumping energy is required; hence the pumping cost would increase.

3. Production loss when the system is shut down to remove the fouling material and do the cleaning.

Cleaning the heat transfer surface from fouling material is needed to restore the efficiency. Depending on the severity of the fouling, frequency of the cleaning process typically differs from a month to a year (Awad, 2011). Production time lost to do the cleaning cannot be recovered.
4. Higher maintenance fee due to the cleaning and using antifoulants.

The cleaning process is not as easy as it seems to be. Cleaning may require that the exchanger be dismantled and reassembled which can result in damage to the heat exchanger. Repairs may be needed due to these damages. Using antifoulants in the system is also common to prevent fouling process. All these mentioned above need more money expenditures on maintenance.

The economic cost of crude oil fouling in the preheat train is huge. It was estimated around 1.2 billion US$ per year (Machietto et al., 2011). It should be noted that removal and disposal of the fouling material and extra emission of CO$_2$ would also cost. CO$_2$, considered as the main greenhouse gas contributing to global warming, is arguably released to the environment due to human activities such as combustion of fossil fuels in transportation, for domestic and industrial heat requirements, and for the production of electric power generation. (Müller-Steinhagen et al., 2009)

Researchers have not quantitatively studied the amount of CO$_2$ discharged to the environment due to fouling. There are some data showing the significant impact of heat exchanger fouling on the environment. About 10 percent of the CO$_2$ emission of refineries to the environment is reportedly due to fouling (Müller-Steinhagen et al., 2009). Calculations based on the amount of oil refining in a year, shows that 2.5 percent of CO$_2$ emissions in a year comes from fouling in the refineries. This number keeps increasing as heavier crude oils need to be processed and the refining procedures become more complex.

The cost of preheat train fouling in one 160,000-bbl/day Total refinery was estimated to be around 1.5 million US$ in 2003 in a three months period. A 200,000-bbl/day UK refinery
reported that a 1°C loss in preheat train oil exit temperature costs around 250,000 £ per year. Other reports suggest that the energy equivalent of 0.25% of all oil production is lost to fouling in preheat train, which is close to 1 day’s production lost per annum (85 million barrels on a worldwide basis) (Macchietto et al., 2009).
Chapter 2: Literature review

2.1 Fouling

Fouling generally represents the accumulation of unwanted and undesired material (dirt, scale, corrosion products) on a surface. This phenomenon is very common in heat transfer processes on heat exchanger surfaces. The presence of this unwanted material on heat exchanger surface is associated with two major issues (Figure 2.1):

1. The fouling layer acts as a further resistance to heat transfer. In general thermal conductivity of this layer is very much lower than that of metals. As a result the heat transfer rate decreases.

2. As the fouling layer becomes thicker, the flow area shrinks and at the same time the roughness of the surface increases, which leads to an increases in pressure drop through the heat exchanger.

![Figure 2.1 Heat transfer coefficient decreasing with time and pressure drop increasing with time](image-url)
These two issues both represent additional energy requirements. The loss of heat transfer efficiency usually means that additional energy must be supplied somewhere else in the system. The increased pressure drop needs an increase in pumping energy requirement to be compensated to maintain the flowrate. With the time passing the fouling could become severe and thermohydraulic performance of heat exchanger degrades. Figure 2.2 shows the fouled surface of tubes in a heat exchanger.

![Fouled surface of the tubes in a heat exchanger](image)

**Figure 2.2 Fouled surface of the tubes in a heat exchanger**

There are also other issues which may not be obvious results of fouling but still are more or less influenced by fouling. They usually fall into two categories; corrosion and additional maintenance. The presence of fouling layers in some cases can protect the metal surface from the effects of corrosion, but more often increases corrosion. An example could be high temperature heat transfer in which heat is being removed from combustion products. High chlorine content of the combustion material can lead to corrosion problems. It should be mentioned that the ash deposit on the heat transfer surface is often rich in active chlorine and can cause rapid corrosion. Disposal of the deposits is an issue due to the environmental concerns.

### 2.1.1 Classification of Fouling Process

Generally fouling types are categorized based on the different physical and chemical processes involved. The five primary categories identified by Epstein(1983) are:
1. Particulate fouling is the accumulation of suspended particles on the heat transfer surface which could be called sedimentation fouling if settling by gravity prevails. This type of fouling could be influenced by the concentration of suspended particles, fluid flow velocity, temperature of the heated surface, and the heat flux applied on the heat exchanger surface.

2. Crystallization fouling which is divided into two subcategories:
   a. Precipitation fouling. It is also called scaling refers to crystallization of the dissolved substances on the heat transfer surface due to solubility changes with temperature.
   b. Solidification fouling: means freezing of a pure liquid or in a multicomponent mixture freezing of the higher melting component onto a subcooled surface.

   In this type of fouling the controlling mechanism is the primary nucleus formation, since the most important phenomenon is crystal growth around this primary nucleus.

3. Chemical reaction fouling occurs as a result of chemical reactions in which the surface material itself is not a reactant. However, the heat transfer surface may act as a catalyst for some reactions such as coking, cracking, polymerization, and autoxidation. This type of fouling is commonly seen in many applications of process industry such as petrochemical industry, oil refining, and cooling of gas and oil. Thermal instability of the chemical species is the main reason for chemical reaction fouling. This type of fouling could occur over a wide temperature range from ambient temperature to 1000°C but is more likely to happen at higher temperatures.

4. Corrosion fouling involves a reaction between the heat transfer surface and the fluid stream. The attachment of corrosion products on the heat transfer surface is the result.
5. Biological fouling may occur due to the attachment and growth of microorganisms and/or macro-organisms on the heat exchanger surface. This problem is mainly associated with water streams and is also called biofouling. Biofouling is categorized in two different subcategories: Microbial fouling and Macrofouling. The first one is accumulation of microorganisms while the second one represents accumulation of macro-organisms. Biological fouling forms a biofilm or a slime layer on the surface that is uneven and difficult to remove.

Epstein (1983) names five sequential steps in fouling process regardless of the type of fouling:

1. Initiation period which refers to the time period before any appreciable fouling, generally at the very beginning of the experiment on a clean heat transfer surface.

2. Transport represents the stage in which the fouling species or a crucial reactant is transported from the bulk fluid to the heat transfer surface.

3. Attachment is the next step in which the transported components stick to the wall where deposit is actually formed.

4. Removal of the deposits may start right after deposition has started or occur later as large portions of deposit flake off. Different models are proposed based on either of these assumptions.

5. Aging, which may include changes in crystal or chemical structure, is the last step which starts as soon as the deposit lays down on the heat transfer surface. Aging may either make the deposit stronger or weaker.
Having a summary of different types of fouling, it should be noted that the main causes of petroleum fouling are the suspended insoluble particles and corrosion products (particulate and corrosion fouling) and chemical reactions (e.g. polymerization, coking, etc.) which can occur in the bulk, thermal boundary layer, or on the surface (chemical reaction fouling).

2.1.2 Fouling resistance measurement

In section 1.1.1 the effect of fouling was discussed. To reduce this economic effect one should measure the fouling resistance as well as fouling rate to know how much fouling occurs over time. Various methods can be used to monitor and measure the fouling thermal resistance. These may be divided into two main categories of analytical and experimental methods, indicated below:

1. Direct weighing; the simplest method is to weigh the heat transfer device before and after fouling to assess the extent of deposition. Using thin walled tube is necessary to reduce the tare mass to increase the accuracy.

2. Thickness measurement; in many cases the thickness of the fouling layer is less than 50 microns which makes it difficult to do the measurement directly. Using a removable plate the thickness measurement can be made using a micrometer or travelling microscope.

3. Heat transfer measurement; as discussed before, deposition of the fouling material results in changes in heat transfer. The basis of this method is using the following equation:

\[ R_f = \frac{1}{U} - \frac{1}{U_{clean}} \]  \hspace{1cm} (2.1)

Where \( R_f \) is the fouling resistance, and \( U \) is the overall heat transfer coefficient. Since this is the method used in the thesis, it will be discussed thoroughly later in this work.
4. Pressure Drop; it is also possible to use the changes in pressure drop. As mentioned before, the pressure drop increases with fouling increasing due to the area shrinkage. It should be noted that roughness of the fouling layer also causes pressure drop. In other words, increasing pressure drop could be an indicator of fouling, but not a good criterion for fouling measurement. In some cases pressure drop increases while there is no significant fouling. This pressure drop is due to the roughness increase. Figure 2.3 shows the latter case.

![Diagram showing fouling resistance and pressure drop over time](image)

**Figure 2.3** Pressure drop increases with time due to the deposit roughness while fouling rate is not significant (fouling resistance is almost zero and is not changing significantly)

### 2.1.3 Heat transfer measurement

As mentioned in section 2.1.2 there are several analytical and experimental ways to measure the fouling resistance. The present work is based on the Heat Transfer Measurement method; one of the experimental methods based on the changes in heat transfer during the deposition process of the fouling material. The set of equations below shows the relation between heat transfer coefficient and the fouling resistance on the inner wall of the heat exchanger: (No matter in a tube or another type of heat exchanger,
these equations are general and applicable, as the geometry is accounted for in the term

A)

\[ Q = UA_i \Delta T_m \]  \hfill (2.2)

\[ \frac{1}{U_{clean}A_i} = \frac{1}{h_i A_i} + \frac{R_{wall}}{A_{wall}} + \frac{1}{h_o A_o} \]  \hfill (2.3)

\[ \frac{1}{U_{fouled}A_i} = \frac{1}{h_i A_i} + \frac{R_{wall}}{A_{wall}} + \frac{1}{h_o A_o} + \frac{R_f}{A_i} \]  \hfill (2.4)

\[ R_f = \frac{1}{U_{fouled}} - \frac{1}{U_{clean}} \]  \hfill (2.5)
This is for thin deposits where, according to the equations above, the fouling resistance at any time equals the inverse of overall heat transfer coefficient at that time minus the inverse of overall heat transfer coefficient at time zero (when the surface is clean). The relation between thermal fouling resistance and the deposit thickness and the mass of deposit per unit surface is expressed as follows:

\[ R_f = \frac{x}{k_f} = \frac{m'}{\rho_f k_f} \]  \hspace{1cm} (2.6)

Where \( x \) is deposit thickness, \( m \) is the mass of deposit per unit surface area, \( \rho_f \) is the density of the foulant and \( k_f \) is the thermal conductivity of the foulant.

Using this method requires the hydrodynamic of the fluid flow to be independent of presence of the deposit. Thus all changes in \( U \) from \( U_c \) are attributed to fouling. Running the experiments under turbulent (and also transition) flow regime will decrease importance of the deposit effect.
on hydrodynamics of the fluid. In the first stages of the deposition, rough surface of the deposit increases the turbulence, though.

Bringing attention to section 2.1.1, adhesion is one of the steps in fouling process and the role of flow parameters such as Velocity, Reynolds number, and wall shear stress on adhesion is undeniable. Therefore there are correlations relating fouling rate to these parameters. In general, the fouling rate for an oil is a function of composition, temperature of surface and bulk fluid, and flow.

\[
\frac{dR_f}{dt} = a_1 f_1(C). f_2(T_s, T_b). f_3(\text{flow rate}) \tag{2.7}
\]

2.2 Flow in an annulus

Having annular shape equipment in industry makes the flow study in an annulus an interesting subject. Researchers have done many investigations on the fluid flow through an annulus. For fouling, examination of deposits is convenient when using an annular hot section with deposit on the inner rod, as the deposit is accessible for examination of structure. To interpret laboratory fouling rate data, the flow field should also be known and preferably in a range that can be applicable to industrial equipment. However, the drawback of using annular shape equipment is the analysis of the flow in it, which is much more complicated than in a circular tube or between two parallel plates. The main reason could be the non-linear radial variation of the total shear stress. With the aspect ratio (inner diameter divided by outer diameter) increasing towards unity the flow is more like the flow between parallel plates, and for aspect ratios close to zero the flow is similar to that in a circular tube. Figure 2.5 shows the
velocity and shear stress distribution in an annulus for laminar flow (Re<2000), where the Reynolds number is based on $d = d_{out} - d_{in}$.

![Fluid flow pattern inside an annulus in laminar conditions](image)

**Figure 2.5 Fluid flow pattern inside an annulus in laminar conditions (Meter and Bird, 1961)**

There are two boundary layers forming from the pipe wall to a point of maximum velocity in fully developed annular flow. Due to the different degrees of curvature of the inner and outer pipe walls, the boundary layers are of different thicknesses and the interaction between these boundary layers results in an asymmetric flow pattern. As a result of this asymmetry, the maximum velocity is located closer to the inner wall than the outer wall.
Kaneda et al. (2003) have studied flow patterns for turbulent flows (Figure 2.6). The dashed line represents the eddy viscosity effect on the wall shear stress.

### 2.2.1 Wall shear stress calculation for fully developed laminar flow

Fully developed laminar flow inside smooth vertical annuli yields asymmetrical velocity profile as shown in Figure 2.6. Solving the equation of motion, the well-known following expression for the flux of momentum is given in r-direction:

$$
\tau = \frac{r_{\text{out}} \Delta p}{2L} \left( \frac{r}{r_{\text{out}}} - \lambda^2 \frac{r_{\text{out}}}{r} \right)
$$

where $\Delta p$ is the pressure drop across the annulus of length $L$ and $\lambda$ is the radius ratio ($r_{\text{max}}/r_{\text{out}}$) at which the velocity is maximum. The shear stress distribution is also shown in Figure 2.6. For laminar flow we have:
\[ \tau = -\mu \frac{dv}{dr} \]  

(2.9)

Insertion of this expression into equation 2.9 and integration will give a velocity distribution of as follows:

\[ v = \frac{r_{\text{out}}^2 \Delta p}{4 \mu L} [1 - \varepsilon^2 + \frac{1 - (r_{\text{in}}/r_{\text{out}})^2}{\ln \left( \frac{r_{\text{out}}}{r_{\text{in}}} \right)} \ln(\varepsilon)] \]  

(2.10)

Where \( \varepsilon = \frac{r}{r_{\text{out}}} \). The average fluid bulk velocity across the annulus will be:

\[ \Delta p = \frac{v_{av} 8 \mu L}{r_{\text{out}}^2 [1 + (r_{\text{in}}/r_{\text{out}})^2 - \frac{1 - (r_{\text{in}}/r_{\text{out}})^2}{\ln \left( \frac{r_{\text{out}}}{r_{\text{in}}} \right)}]} \]  

(2.11)

Thus by calculating average velocity from the average volumetric flow rate, Equation 2.11 gives the pressure drop, from which the shear stress can be calculated. Since in the annular fouling apparatus the inner wall is heated and the deposit is forming on it, the effect of inner wall shear stress on fouling rate is investigated, and substituting \( r \) by \( r_{\text{in}} \) in equation 2.8, yields \( \tau_{\text{in}} \)

\[ \tau_{\text{in}} = \frac{r_{\text{out}} \Delta p}{2L} \left( \frac{r_{\text{in}}}{r_{\text{out}}} \right)^2 \left( \frac{r_{\text{in}}}{r_{\text{out}}} \right)^2 - \lambda^2 \]  

(2.12)

### 2.2.2 Maximum Velocity and Zero Shear Stress in an Annulus in turbulent flow

The early researches on the flow of Newtonian fluids by Knudsen and Katz(1958), Rothfus et al.(1955), Walker and Rothfus(1958), Brighton and Jones(1964) were based on the coincidence of the positions of maximum velocity and zero shear stress (Japper-jaafar et al.,2010), as for example in a circular duct. Lorenz (1937) discovered experimentally that in
turbulent flow in an annulus, the location of maximum velocity is different from that for laminar flow. Kjellstrom and Hedberg (1966) derived an integral expression from which the local shear stress could be achieved. Based on this expression the location of the maximum in the velocity differs from that of the laminar flow and also from the location of the zero in the total shear stress. They could not prove it experimentally for a smooth annulus, but experiments on an annulus with a roughened inner surface confirmed the idea. Lawn and Elliott (1972), using hot wire anemometry, showed the non-coincidence for smooth annuli. Their results pointed out that for radius ratios less than 0.4 the position of zero shear stress was closer to the inner pipe wall than that of the maximum velocity. They also found the inner velocity profile different from the one derived from pipe-flow data. Later on, Rehme (1974) also approved their findings by studying fully developed turbulent flow of air using hot wire anemometry in concentric annuli with different diameter ratios (0.02, 0.04, 0.1). Nouri et al. (1994) used Laser Doppler anemometry for both Newtonian and non-Newtonian fluid (Re>8900 for Newtonian and Re>1150 for non-Newtonian fluids). They could not find the location of zero shear stress distinguished from that of the maximum velocity. However, they found these positions to be closer to the inner pipe wall, and independent of the Reynolds number. Chung et al. (2002) also confirmed that the positions of zero shear stress are closer to the inner wall than those of the maximum velocity using Direct Numerical Simulations of turbulent concentric annular flow for two different diameter ratios of 0.1 and 0.5 at Re=8900.

2.2.3 Transitional annular flow

Rothfus et al. (1955) investigated air flowing at moderate Reynolds number range (1250<Re<21600) through annuli with two different diameter ratios. Based on their results the position of maximum velocity is the same for laminar and turbulent flow in both annuli. But for
the transitional regime the position is closer to the inner pipe. It is also noted that the Reynolds-number range for the transitional regime was wider and more dependent on diameter ratio compared to pipe flow.

A similar procedure was done on water by Rothfus and Walker (1958) to find the radial location of maximum velocity. The results show that for fully-turbulent flow and laminar flow this location is the same. In the early transitional regime location of the maximum velocity starts to shift towards the inner pipe and as Reynolds number increases, it shifts backward and at a critical Reynolds number of 2200 stops changing. For larger Reynolds number this location re-approaches its laminar value.

2.2.4 Theoretical analysis of turbulent flow in an annulus

A theoretical analysis by Hanks and Bonner (1971) on the stability of laminar flow in a concentric annulus predicts that the inner flow region is less stable compared to the outer one. In other words, when the flow in the inner flow region undergoes transition to turbulent, the flow in the outer region remains laminar. The significant changes in the momentum transport mechanism to a turbulent mode increase the inner wall shear stress which results in a shift in the radial location of the maximum velocity towards the outer wall. When the outer region flow undergoes transition to turbulence the radial location of the maximum velocity reaches its maximum and then decreases to a value below that of laminar flow.

2.2.5 Mathematical modeling of turbulent flow in an annulus and shear stress calculation

The mathematical modeling of turbulent flow in annuli includes the correlations to define the relation between wall shear stress, pressure drop and friction factor. The general idea is the dependency of pressure drop on three non-dimensional groups; Reynolds number, friction factor, and a geometric factor a (Meter and Bird, 1961), and the relation between pressure drop and total
wall shear stress. The proposed correlations are different depending on how Reynolds number, geometric factor, and friction factor are defined. Table 2.1 shows some of the proposed mathematical models.
Table 2.1 Mathematical models proposed for annular friction factor and shear stress in transitional and turbulent regimes by different researchers

<table>
<thead>
<tr>
<th>Researcher</th>
<th>Formula</th>
<th>Fanning Friction Factor ( f(F) )</th>
<th>Shear Stress Formula</th>
</tr>
</thead>
<tbody>
<tr>
<td>Jones &amp; Leung</td>
<td>( \frac{\rho V D_t}{\mu} )</td>
<td>( \frac{1}{\sqrt{f}} = 2 \log(Re \sqrt{f}) - 0.8 )</td>
<td>( \frac{f(D)}{8 \rho V^2} )</td>
</tr>
<tr>
<td>Meter &amp; Bird</td>
<td>( \frac{\rho V D_t}{\mu} )</td>
<td>( \frac{1}{\sqrt{f}} = 4 \log(Re(a) \sqrt{f}) - 0.4 )</td>
<td>( \frac{f(F)}{2 \rho V^2} )</td>
</tr>
<tr>
<td>Fredrickson &amp; Bird (1)</td>
<td>( \frac{\rho V D_{out}}{\mu} \left( \frac{1 + a^2}{(1-\alpha)^2} - \frac{1 + \alpha}{\ln(\frac{1}{\alpha})} \right) )</td>
<td>( f = 0.0791/Re^{0.25} )</td>
<td>( \frac{f(F)}{2 \rho V^2} )</td>
</tr>
<tr>
<td>Fredrickson &amp; Bird (2)</td>
<td>( \frac{\rho V D_{out}}{\mu} (1 - \alpha) )</td>
<td>( f = 0.0791/Re^{0.25} )</td>
<td>( \frac{f(F)}{2 \rho V^2} )</td>
</tr>
<tr>
<td>Kaneda</td>
<td>( \frac{\rho V D_h}{\mu} \left( \frac{2}{f_{wm}} \right)^{1/2} = 1.610 + \frac{1}{0.436} \ln{Re} - \frac{550}{Re\left(\frac{f_{wm}}{2}\right)^{1/2}} - \frac{1}{0.436} \ln\left(\frac{2}{f_{wm}}\right)^{1/2} )</td>
<td>( f(F) )</td>
<td>( \frac{f(F)}{2 \rho V^2} )</td>
</tr>
</tbody>
</table>

* \( f_{wm} \) in Kaneda’s equation stands for the friction factor based on mean wall shear stress. \( f(D) \) is the Darcy friction factor which is four times bigger than Fanning friction factor.

** \( D_t = D_k \varphi_a \cdot \varphi_a = \frac{1}{(1-a)^2} \left[ 1 + a^2 - \frac{1-a^2}{\ln^2 a} \right] \)

*** \( D_h = D_{out} - D_{in} \)

These correlations are developed under transition and fully developed turbulent regime.

Figure 2.7 shows comparative prediction of wall shear stress for the five cases of Table 2.1.
Figure 2.7 Wall shear stress versus $Re(a=0.42)$ according to the equations listed in Table 2.1

The inconsistency of the curves is due to different methods of calculation. (Either in calculating the Reynolds number from the hydraulic diameter or the correlation developed for Fanning friction factor and shear stress).

Kaneda’s correlation is based on an investigation using a combination of theoretical and correlative methodologies and seems to be more accurate. More precisely, he first develops improved theoretically-based correlating equations for the turbulent shear stress distribution. He then uses these expressions to calculate total shear stress. Then this expression of total shear stress is used to achieve correlations for other flow parameters such as time-mean velocity. In mathematical structure to represent turbulent flow, the basic equation is to write wall shear stress as a function of viscosity, derivative of the velocity, and fluctuating velocity parameter. However, this is the total wall shear stress. Shear stresses of the inner and outer walls do not
necessarily have equivalent values. But there is a relation between these two based on the diameter ratio. The following equation represents the relation between shear stresses.

\[
\frac{\tau_{in}}{\tau_{out}} = \frac{r_2}{r_1} \frac{\left( r_0^2 - r_1^2 \right)}{\left( r_2^2 - r_0^2 \right)}
\]

\[
\tau_m = \frac{r_1 \tau_{in} + r_2 \tau_{out}}{r_1 + r_2}
\]

Having \( r_1 \) and \( r_2 \) as inner and outer radius, \( r_0 \) as the radius at which the wall shear stress is zero, and \( \tau_m \) as mean shear stress on the two walls. Here, the shear stress on the inner wall is of most interest, as the deposit forms on the inner heated rod.

2.3 Fouling of oils caused by asphaltene deposition

Crude oil, a complex mixture of hydrocarbons, is divided into several classes on different bases. In terms of polarity and molecular mass it is divided into four chemical classes: Saturates, Aromatics, Resins, and asphaltenes. Asphaltenes are the heaviest and most polar class, insoluble in saturates such as n-heptane or n-pentane and soluble in aromatics such as toluene. The high molecular weight of this fraction, which usually falls into 2000(\pm)500 Da, and its polar nature results in its tendency to form another phase in its lower molecular mass, non-polar surroundings and precipitate. There are still researches going on chemical structure and physiochemical properties of this fraction, because of its complexity. Based on the plot of molecular weight versus hydrogen content of petroleum provided by Wiehe (1999), high aromaticity and molecular weight of asphaltenes result in insolubility of this chemical class in n-heptane.
2.3.1 Deposit formation by asphaltenes

Field experience of De Boer et al. (1995); Kokal and Sayegh (1995) and some experimental observations of (Andersen, 1994; Hammami et al., 2000; Thomas et al., 1992) show that deposition of asphaltene is dependent on various factors including the composition of the surrounding fluid, pressure and temperature (Mitchell and Speight, 1973).

How the temperature impacts the asphaltene deposition is a controversial subject in the literature. Lambourn and Durrieu (1983) in a research on three oils observed interesting behavior of its deposition at elevated temperatures. For the first oil asphaltenes completely dissolve in the temperature range 100-140°C but re-precipitate as the temperature exceeds 200°C. For the other two oils the solubility either increases or does not change with temperature increasing. Storm et al. (1996) also supports the view of re-precipitation at higher temperatures by doing rheological studies on asphaltene flocculation. Others claim that asphaltene solubility increases monotonically with temperature.

It is believed that pressure and composition have greater impact on asphaltene precipitation compared to temperature. Pressure drop can also destabilize asphaltenes. Depressurization during oil production decreases the density of the crude oil, as a result the screening effect of other components of the oil on asphaltene interactions decreases, causing the interactions between asphaltenes to become stronger which causes the precipitation.

To see the effect of composition on asphaltene precipitation Asomaning and Watkinson (2000) did some experiments in which the concentration of asphaltene was increased by increasing the heavy oil content of the fluid. Despite the expectations, the deposition rate decreased, suggesting that it is not the concentration of asphaltene alone that has an impact on its
deposition. This is how instability of the oil, tendency of the oil to form another phase, was known to be the driving force for asphaltene deposition.

2.4 Stability criteria

The stability of different oil mixtures could be predicted using two approaches which are closely related; Andersen/Pedersen Model and Wiehe’s Oil Compatibility Model. These approaches relate to the situation where a volume $V_o$ of an asphaltene containing oil is diluted in a volume $V_s$ of solvent (eg. toluene) and then a precipitant (eg. heptane) is added to the mixture.

2.4.1 Andersen/Pedersen Model

According to this approach asphaltene precipitation starts when the average solubility of the solvent phase equals the critical solubility parameter. The critical solubility parameter for the mixture is as follows:

$$\delta_{cr} = \varphi_o \delta_o + \varphi_p \delta_p + \varphi_s \delta_s$$  \hspace{1cm} (2.15)

Where $\delta_o$, $\delta_p$, and $\delta_s$ are the solubility parameters of oil, precipitant and solvent respectively and $\varphi_o$, $\varphi_p$, and $\varphi_s$ are volume fractions of corresponding components. So if three precipitation tests are done with different ratios of $Vol_p/Vol_o$ and $Vol_s/Vol_o$ a plotting of $Vol_p/Vol_o$ vs $Vol_s/Vol_o$ would give us a line with the slope of $(\delta_s - \delta_{cr})/(\delta_{cr} - \delta_p)$, and the intercept of $(\delta_o - \delta_{cr})/(\delta_{cr} - \delta_p)$. As long as $\delta_o$ is greater than $\delta_{cr}$ the oil blend is stable and no precipitation happens.

2.4.2 Wiehe’s Model

In the Oil Compatibility Model (OCM) of Wiehe the critical solubility parameter is expressed as

$$\delta_f = \varphi_o \delta_o + \varphi_H \delta_H + \varphi_T \delta_T$$  \hspace{1cm} (2.16)
Where $\delta_f$ represents the flocculation solubility and $\delta_o$, $\delta_H$, and $\delta_T$ are respectively the solubility of oil, n-Heptane (precipitant) and Toluene (solvent).

For this method the slope and intercept for the flocculation titration plot are as follows:

$$S_{BN} = 100 (\delta_o - \delta_H)/(\delta_T - \delta_H) \quad (2.17)$$

$$I_N = 100 (\delta_f - \delta_H)/(\delta_T - \delta_H) \quad (2.18)$$

$S_{BN}$ and $I_N$ are respectively solubility blending number and insolubility number.

For mixtures,

$$S_{BN_{mix}} = \frac{Vol_1 S_{BN1} + Vol_2 S_{BN2} + Vol_3 S_{BN3} + \cdots}{Vol_1 + Vol_2 + Vol_3 + \cdots} \quad (2.19)$$

The latter equation suggests that the solubility parameter of a mixture is the volumetric average solubility parameter. Besides if the oil contains no asphaltene which means is completely soluble in n-Heptane the insolubility number is zero but if the asphaltene-resin dispersion is barely soluble in toluene, the insolubility number is 100.

For a mixture of oils the solubility of the mixture must be higher than flocculation solubility parameter of any oil in the mixture for mixture compatibility. In other words the compatibility criterion is $S_{BN_{mix}} > I_{N_{mix}}$.

Hong E. and Watkinson (2005) showed that fouling rate can be correlated either by the Anderson/Pederson model, or the Wiehe model, and that the key parameters of both models are related.
2.4.3 Colloidal instability index

The Colloidal Instability Index (CII) treats the crude oil as a suspension made of saturates, aromatics, resins, and asphaltenes. This index is an expression of the colloidal nature of the suspension which is the mass ratio of the sum of the asphaltene and saturates which precipitate asphaltenes to the sum of the aromatics which dissolve and resins that peptize asphaltenes as follows:

\[ CII = \frac{(Saturates + Asphaltenes)}{(Aromatics + Resins)} \]  (2.20)

SARA analysis gives the weight percentages needed for colloidal stability index. CII has been used to determine the stability of asphaltenes in crude oil solvent blend by Watkinson and Asomaning (2000). The oils with CII of 0.9 and higher are known as oils with unstable asphaltene and CII of 0.7 and lower indicates the oils with stable asphaltene, and between 0.7 and 0.9 there is an uncertainty on the stability of asphaltene, reported Asomaning (2003). The comparison of the performance of CII and other criteria, such as asphaltene-resin ratio, Oliensis Spot Test number and the asphaltene Precipitation Detection Test (APDT) number from the titration method, in their work showed that the prediction of CII on asphaltene precipitation is more precise. Moreover CII is calculated through SARA analysis which is relatively easy to do. However using CII is not of any help when the asphaltene concentration is very low in the oil.

2.5 Asphaltene precipitation by composition changes

Paraffinic compounds, due to their solvent power, affect the interaction between resins and asphaltenes. This is the reason why addition of paraffinic compounds to the oil changes the asphaltene solubility. In cases where the paraffinic compound is a good solvent of resins and not that of asphaltene, with volume of the diluent increasing the interaction between resins and asphaltene and also the oil capacity to stabilize the asphaltene gets weak and then asphaltene
precipitates. Mitchell and Speight (1973) used a range of nonpolar solvents and solvent blends to precipitate asphaltene from Athabasca bitumen. Based on their results, the amount of asphaltene that precipitates upon the addition of solvent changes linearly as the solubility parameter of the solvent. The yield of the precipitate of each solvent depends on the difference that the solubility parameter of asphaltene and the solvent have.

Buenrostro and Lira-Galeana (2004) did also some experiments to investigate the effect of composition changes on asphaltene precipitation. They used dead-oil samples taken from surface equipment of two problematic Mexican oil wells. The compositions of these oils were defined in previous works. Considering Wiehe’s method, which was discussed in section 2.4.2, they used n-alkanes to determine the amount of precipitated asphaltene as a result of oil titration using n-alkanes. The experiment consists of adding a volume of n-alkane to the oil in a flask, 10 minutes of ultrasonic shaking of the flask, leaving the sample overnight, followed by vacuum filtration using a 0.45 micron Teflon filter, drying the filter and finally weighing it. Figure 2.8 is a summary of this experiment:

**Figure 2.8** Crude oil titration experiment, Buenrostro and Lira-Galeana (2004)
Their results show that for a given dilution ratio, with number of carbon increasing the amount of asphaltene precipitated decreases and the precipitation increases as the solvent/oil ratio increases for concentrations less than 10 $cm^3$ of n-alkane/g of oil. For the higher concentrations
precipitation increases by very small amounts. These results are in agreement with what
Hirschberg et al., 1984; Kokal et al., 1992; and Rassamdana and Sahimi, 1996 have reported.

2.6 Mechanism for asphaltene deposition

Asphaltene precipitation in oil refineries is found to be a physical process. Deposit
formation on heat transfer surface could be a physical process, chemical reaction, or a
combination of both though.

Dickakian and Seay (1988), believing deposit formation is based on the incompatibility
between asphaltene and other components of crude oil, summarized the mechanism in three
steps; Precipitation of asphaltene, adherence of precipitated asphaltene to heat transfer surface,
and coking of asphaltene on the surface. This model describes asphaltene deposition as a physio-
chemical process, considering precipitation as a physical process, coking which only occurs at
elevated temperatures (T>360°C) as a chemical reaction, and adherence as a physio-chemical
step.

Lambourn and Durrieu (1983), finding asphaltene precipitation in the crude oil bulk the
main reason of fouling, proposed a mechanism which could be used only when water and
particulates are present. This mechanism includes: asphaltene precipitation in the bulk, formation
of a stable emulsion as a result of interactions of the asphaltenes, water, and particulates, and the
deposition of the emulsion on the heated surface followed by aging.

Eaton and Lux (1984) proposed a fouling mechanism based on a series of chemical
reactions including polymerization and condensation in the presence of oxygen. Based on this
model under heating conditions, resins and aromatics can be converted to asphaltenes. Thus,
when crude oil is exposed to heat, the concentration of asphaltene increases. These chemical
reactions happen at very high temperature (400-500 °C).
Asomaning’s (2000) proposed mechanism based on experimental data consists of the following steps: asphaltene in the fluid could either aggregate or solubilize, depending on the composition of the fluid. This step is correlated by the Colloidal Instability Index. There are filterable aggregated solids formed by asphaltene with a composition similar to it, and with temperature increasing the concentration of insoluble asphaltene decreases which results in a decrease in fouling. Adhesion of asphaltene solids on heated surface is an important step considering the high activation energy and negative velocity effect. The final step is aging of asphaltene solids to form a coke-like deposit different from the original asphaltene.

2.7 Objectives of the present work

The general objective of this work was to carry out experiments to investigate the effect of wall shear stress on the fouling rate in an annular geometry under asphaltene precipitation. However, to reach the objective, different specific elements were identified:

- Review of literature on petroleum oil fouling, focusing on oil composition and fluid flow effects
- Selection of a convenient system for asphaltene precipitation fouling study
- Improvement of the existing fouling loop with new equipment and data acquisition system
- Development, testing and analysis of heat flow in two new annular heater probes
- Calculation of wall shear stress effects in current and former annular probe fouling studies
- Relation of fouling rate to calculated wall shear stress and other process variables
Chapter 3: Experimental apparatus and procedure:

3.1 Experimental apparatus

The fouling experiments were performed using a circulation loop which consists of three main parts: a feed tank, a 1-1/2 HP centrifugal pump, and an annular heating section. Figure 3.1 shows a schematic diagram of the fouling loop. Although basic parts of the pump have been used before, a new pump, three new heating probes, and other modifications were made for the present work.

3.1.1 Feed Vessel

A 9.45 L, 316 stainless steel tank (203mm ID * 292mm L) holds the oil sample. The temperature of the liquid inside the feed tank is maintained constant using a stainless steel cooling/heating coil (9.5 mm OD tubing) in which either cooling water (V13) or low pressure steam (V12) flows. Thermocouples are installed at cooling water inlet and outlet, liquid tank, liquid inlet and outlet to the heating section to measure the temperature. Two parallel heating tapes (each provides 1200 watts of heat) are wrapped around the tank just in case to be used when the steam heat is not enough to keep the liquid temperature at the desired level. To provide the required pressure and to check for any leakage in the system, compressed \( \text{N}_2 \) and air or a mixture of both gases are used. The gas could be supplied to the tank either from the top (through the gas phase of the tank) or could be bubbled from the bottom (through the liquid phase) to eliminate any dissolved oxygen. Three check valves are installed at the gas inlet to prevent the back flow. A pressure transducer is placed in the tank to measure the vapor phase pressure of the liquid tank. Valves 15 and 16 are used for liquid sampling during the flow circulation and preheating respectively. To minimize the total heat loss of the system all the
pipes are wrapped using insulation material. The system was totally checked for any kind of leak (gas or liquid) before installation of the insulation.

3.1.2 Pump and Flow Measurement device

A 1.5 HP centrifugal Packless (401A-8879) pump with maximum frequency of 90 Hz and maximum flow of 27 gpm circulates the fluid through the pipes, passing it through the annular heating section returns it to the feed tank. This pump was installed as part of this work. Standard municipal clean water is used to cool down the pump. An orifice plate and a differential pressure transducer (Model PX771-100DI, OMEGA Engineering Inc.) are used to measure the flow rate. The orifice plate was calibrated over the Reynolds number range of 3000 to 8000 using Paraflex (an organic product of the Petro-Canada), resulting in a discharge coefficient of 0.655 which was used to calculate the flow rate based on the pressure difference measured during the experiments. The desired flow rate is obtained by adjusting the liquid bypass valve (V10) and liquid return valve (V11).
V-1 and V-2: Nitrogen and Air inlet valves; V-3: Gas inlet; V-5: Gas sparging; V-6: Gas (vapor phase); V-8: Venting valve; V-9: Liquid Tank shut-off valve; V-10: Liquid bypass valve; V-11: Liquid return valve; V-12: Cooling water valve; V-15: Liquid sampling valve; V-16: Liquid discharge valve.
DPM: Differential pressure monitor

Figure 3.1 The fouling loop setup
3.1.3 Heater element

An electrical heating element (Gordo Sales Inc., Texas), made of SS 316, with total heating area of $3.4 \times 10^{-3} \, m^2$ (0.95 cm diameter and 11.4 cm heating length) is used. The maximum heater density is $36 \, W/cm^2$ (1000 W at 120 V). A thermocouple is placed in the center of the cartridge (in both radial and axial position). It is located in a well-defined position so that surface temperature could be calculated as will be shown below or calibrated using the Wilson method. Figure 3.2 shows the heating element sketch, including resistance wires and thermocouple.

![Figure 3.2 Heating element structure with the thermocouple at the center](image)

Figure 3.2 Heating element structure with the thermocouple at the center
3.1.4 Probe #1

The fouling probe uses the heating element of Figure 3.2 mounted in an annular section of some 80 cm length. For Probe #1, 82 cm long, 1.27 cm OD, and 0.85 cm ID SS316 tube was drilled out to a diameter of 1.05 cm and a depth of 35 cm such that the heating element slid down to rest on the shoulder of the original tube. The probe casing was filled with Magnesium oxide particles on the top to protect thermocouple and resistance wires from heat. Figure 3.3 shows the details of the heating probe with the element inside.

As shown in Figure 3.1 the heating probe is held straight in the outer annulus of 2.54 cm OD of the fouling loop using fittings and a connection box on top which connects the resistance wires to DC power supply and has also an output for thermocouple wires. The probe is hung in the outer annulus and does not touch its walls. Figure 3.4 shows the heating probe with its connection box.
Figure 3.3 (a) Overall Probe (b) Detailed sketch of the probe (not to scale)
3.1.5 DC Power Supply

The power to the heating element is provided using a B&K Power Equipment Inc. DC power supply (BK Precision VSP12010) with maximum values of 120 and 10 for voltage and amperage respectively. The VSP series power supplies are variable switch mode power supplies designed to deliver power at constant voltage and constant current. VSP Power Supply achieves an energy conversion efficiency of 80%, while keeping noise levels under 20 millivolts. The generator keeps the voltage at $\pm 0.2\%$ of the nominal value.
3.1.6 Data Acquisition System

To record the data (temperature and pressure), a data acquisition module, OMB-DAQ-2416-4AO produced by OMEGA, with 24-bit analog input for voltage or temperature was installed for this work. It also includes eight digital I/O lines and two counters and four analog output channels with 16-bit resolution. This device receives the signals and converts them into digital numeric values that can be manipulated. Software developed for the fouling loop by Alex Thng is used to track and record all the required data; inlet and outlet temperatures of the fluid to the annular section, the core temperature of the probe, temperature and the pressure of the liquid tank, as well as pump frequency and corresponding fluid velocity, voltage and current to the heating element, and data saving mode (either average or real time) are the trackable properties on the software. All the recorded data are transferred to an excel sheet to be analyzed. The analog output is used to transfer data to a control program to control the heat provided by the DC power supply.

Figure 3.6 Data acquisition module

As the data acquisition system was new, the author worked closely with technician Alex Thng to ensure that the system would accurately monitor the thermocouple.
3.1.7 Safety Associated Measures

To prevent the probe from overheating, safety software is incorporated in the system. If the core temperature exceeds a maximum allowable set-point temperature, the system will shut down the power supply to the probe to prevent any overheating damage of the system. The power supply to the pump should be maintained when the probe power is shut down. Due to the hazardous nature of the crude oil used in the experiments the whole fouling experimental unit is placed into an enclosure box connected separately to the building ventilation system. The liquid pressure and temperature are logged for the alarm in the safety control program.

Considering the long duration of the fouling runs, a local area network is set up for the data-logging computer, so that the operator is able to access the recorded experimental data from off-site in a given time interval. Figure 3.7 and Figure 3.8 show top part of the fouling loop and the pump case respectively.
Figure 3.7 Top part of the fouling unit showing Nitrogen (1) and Oxygen (2) inlets, liquid tank with heating tape (3), the bypass valve (4), the orifice and DP cell (5), and the insulated heating probe (6).

Figure 3.8 The pump case and insulated piping
3.2 Experimental procedure

3.2.1 Sample Preparation and heat transfer fouling runs

Properties of ATB are listed in Table 4.9. It is noted that the viscosity at low temperature is very high. ATB is heated up to 40-50°C to allow it to flow easily. Depending on the desired percentage of ATB in the sample, it is weighed into a 10 l bucket, and a portion of about 6 kg Paraflex at room temperature is gradually added to it while a high shear mixer (Silverson L4RTA, Figure 3.9) mixes the fluids to provide a uniform mixture, while the heater is used to maintain the temperature (40-50°C). The sample is then loaded into the fouling loop feed Tank. The rest of Paraflex at room temperature is added directly to the tank to make a sample of 8 liters.

![Silverson L4RTA high shear mixer](image)

Figure 3.9 Picture of the Silverson L4RTA high shear mixer

3.2.2 Pre-heating, pressurizing the system and software setup

Using the \( N_2 \) valve, nitrogen is purged into the top of the tank. The total pressure of the tank is set at 50 psig (445 kPa, abs). On the software, the maximum pressure and temperature of the tank and maximum temperature of the probe, as well as desired bulk temperature (85°C) is
set. Depending on the desired bulk temperature, heating tape around the tank is used to heat up the fluid. The pump is run first at low frequency (low flow rate). It takes two or three hours for the bulk temperature to be steady.

3.2.3 Probe power-up

When the bulk temperature is 20 to 30°C below the desired value, the power to the probe is switched on. Using the DC power supply the voltage is gradually increased to get the desired core temperature value, after which the heat flux to the probe remains almost constant (a variation of a few percent is normal).

3.2.4 Fouling runs and recording data

Depending on the changes to the probe temperature, duration of runs could differ from one to another. However, experiments are typically carried out for 24 to 48 hours or less if the probe temperature reaches its limit before this time. The software is designed to record data either as real time data or as the average data over a specific time interval. Generally the average data is recorded each minute as a text file, and for further analysis is copied in an excel spreadsheet.

3.2.5 Shutdown procedure

If the system is shut off automatically, all the input power to the pump and to the probe is cut off. Otherwise to end an experiment, first the power to the heating tape is cut and cooling water flow is maximized, then the probe power is decreased gradually to zero (over about 15 minutes) while the pump is still circulating the fluid to cool down the probe. When the probe and bulk temperature drops to 25 to 30°C the pump is shut down gradually. The pressure of the tank is released to atmospheric through the vent valve, and the probe is removed carefully to prevent any deposit from being scraped off the surface. If the deposit is not too soft, the diameter of the
probe with deposit on it is measured using a micrometer. The deposit is then rinsed using n-heptane and allowed to dry naturally before being removed by a soft plastic blade. The probe was cleaned by Varsol and was polished using a fine emery cloth (grit number 300) for the next experiment.

3.2.6 Flushing the loop

The oil sample is completely drained from the system. Air or nitrogen is used to pressure the system slightly to make sure that the oil is completely drained.

3.2.7 Experimental calculation methods

The power of heating probe is kept constant, while the fluid circulates the loop, which results in a constant heat flow, $Q$. The bulk temperature of the fluid is also kept constant using the coil in the tank and the heating tape around it. With the time passing, and fouling happening, the procedure could be formulated as follows:

$$ q = \frac{Q}{A} = U(T_{core} - T_{bulk}) \quad (3.1) $$

$$ R_f = \left( \frac{1}{U} - \frac{1}{U_{clean}} \right) \quad (3.2) $$

$$ = \left[ (T_{core} - T_{bulk}) - (T_{core} - T_{bulk})_{clean} \right] / q $$

$$ = \left[ T_{core} - T_{core,clean} \right] / q $$

Based on these equations with the fouling resistance increasing at constant heat flux and bulk temperature, the core temperature increases. The following graph shows what has been discussed above.
The slope of the changes in surface temperature would give the fouling rate. The experimental procedure is discussed in details as follows.

The amount of heat delivered to the oil is determined by the software from the voltage and current given by the DC power supply. Bulk and probe core temperature are also recorded. Knowing the heat transfer area, the overall heat transfer coefficient is defined as:

$$Q = VI = UA(T_{surface} - T_{bulk})$$

(3.3)

Rewriting the latter equation as a function of time and $Q$ as flux we will have:

$$q_t = \frac{Q}{A} = U_t(T_{surface}(t) - T_{bulk}(t))$$

(3.4)

Figure 3.10 $T_s$, $Q$, and $U$ changing with time
Hence,

\[ U_t = \frac{q_t}{(T_{surface}(t) - T_{bulk}(t))} \]  

(3.5)

Since \( U_{t=0} \) is a constant (calculated from the initial data points), the slope of the curve \( 1/U(t) \) versus time, \( t \) gives the fouling rate.

\[ \frac{dR_f}{dt} = \frac{d}{dt} \left( \frac{1}{U(t)} \right) \]  

(3.6)
Chapter 4: Results

4.1 The effect of diluent on precipitation of ATB

As mentioned before, mixing various crude oils or adding a diluent to a crude oil would change the composition of the fluid. Following mixing, the colloidal instability index may change and the fluid may become unstable, thus causing asphaltene precipitation and fouling. These changes in stability of the oil are due to the changes in interactions between oil constituents. Depending on the chemistry of the diluent the changes in C.I.I are different and consequently the amount of asphaltenes precipitated is different. For rapid fouling in laboratory using an appropriate diluent results in more precipitation and fouling in a short period of time. Moreover, ATB is a very viscous fluid and mixing it with a diluent will reduce viscosity, and generate turbulent flow at a temperature that is close to the preheat train.

4.1.1 A review on available diluents

There are various diluents produced for different purposes. Three were chosen to see how the precipitation process is affected by using diluent. A brief review on these diluents is as follows:

Paraflex HT oils, produced by Petro-Canada are mixtures of hydrotreated and hydrocracked base oils and are used as a diluent or carrier in the production of a variety of finished industrial products. This viscous liquid comes in different HT numbers based on different compositions and consequently different physical properties.

Voltesso is a brand of transformer insulating oil made from base oil and additives and provided by Imperial Oil Ltd. It is not as viscous as Paraflex, and it can also be used as a diluent.

Norpar is a paraffinic hydrocarbon produced by Exxon-Mobil to be used as feedstock, fuel, and solvent. Different numbered Norpars are available in the industry.
Table 4.1 compares some properties of these three diluents. Specific gravities of the diluents are fairly close. Considering the viscosity at two different temperatures, viscosity of Norpar 13 is very low compared to the others which means that we can have lower viscosity for the mixture at lower temperatures. However this fluid is apparently no longer available. So tests were done using Paraflex HT10 and Voltesso 35.

<table>
<thead>
<tr>
<th>Diluent</th>
<th>PARAFLEX</th>
<th>VOLTESSO</th>
<th>NORPAR</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Composition</strong></td>
<td>Mixture of hydrotreated and hydrocracked base oil</td>
<td>Base oil blended with selected additives</td>
<td>Paraffinic hydrocarbons</td>
</tr>
<tr>
<td><strong>Application</strong></td>
<td>Process oils used as a diluent or carrier</td>
<td>Insulating oil used in transformers</td>
<td>Used as feedstock, Fuel, Solvent</td>
</tr>
<tr>
<td><strong>Manufacturer</strong></td>
<td>Petro-Canada Lubricants Inc.</td>
<td>Imperial oil</td>
<td>Exxon-Mobil</td>
</tr>
<tr>
<td><strong>Specific Gravity @15°C</strong></td>
<td>0.857</td>
<td>0.88</td>
<td>0.76</td>
</tr>
<tr>
<td><strong>Special Specification</strong></td>
<td>Stable, reactive with oxidating agents</td>
<td>Stable under normal conditions, high resistance against oxidation and sludge formation</td>
<td>Stable under normal conditions, strong oxidizer</td>
</tr>
<tr>
<td><strong>Physical State</strong></td>
<td>Viscous Liquid</td>
<td>Liquid</td>
<td>Liquid</td>
</tr>
<tr>
<td><strong>Kinematic Viscosity (cSt)</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>100°C</td>
<td>2.8</td>
<td>2.1</td>
<td>NA</td>
</tr>
<tr>
<td>25°C</td>
<td>18.64</td>
<td>18.07</td>
<td>2.4</td>
</tr>
</tbody>
</table>

4.1.2 The effect of using Paraflex as a diluent on asphaltene precipitation

To see the effect of diluent on asphaltene precipitation in ATB, preliminary experiments were done in which different samples were prepared with different ratios of n-Heptane and Paraflex as diluent. N-Heptane is used as a non-solvent of asphaltene to see the effect of Paraflex on asphaltene precipitation in ATB. After blending, the solutions were placed in a dark cabinet
for different time periods (one day and three days), and then using vacuum filtration at room temperature the precipitates are collected. Replication experiments were done to increase the accuracy of the results.

In all samples, ratio of the mixture of n-Heptane and Paraflex to ATB is approximately constant, but different ratios of n-Heptane and Paraflex are used. After a specific time period (either 1 day or 3 days), the samples were heated to 90°C, as bulk temperature in the planned fouling experiments were 80-85°C, and filtered immediately so that the temperature would not drop significantly. The vacuum level during the filtration is approximately constant and around -24 in.-Hg. A Whatman 3 micron Teflon filter is used to collect the precipitate. Filters are dried in an oven (at 100°C) for 10 minutes before being used, to reduce the moisture level in the filter. When the filtration is over, the filters with precipitant were dried in an oven for 15 minutes at 90°C. All the filtration equipment is weighed before and after filtration to determine any precipitate attached to them. Figure 4.1 shows flasks after filtration, which indicates some precipitate is left at the bottom of the flask.

Figure 4.1 ATB+Heptane after one day
The results of these experiments are summarized as follows. The percentage of each component in the sample and the percentage of ATB precipitated in the mixture are presented. The data in Table 4.2 refers to results of filtration at 90°C for one day time period. Table 4.3 shows the results of a three day time period. Table 4.4, and Table 4.5 presents the results of duplicate experiments for time period of 3 days.

**Table 4.2 Component ratio of the samples and the percentage of ATB which precipitated - Time period: 1 day**

<table>
<thead>
<tr>
<th></th>
<th>Solution(1)</th>
<th>Solution(2)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Weight(g)</td>
<td>%</td>
</tr>
<tr>
<td>ATB</td>
<td>2.2</td>
<td>3.6</td>
</tr>
<tr>
<td>Paraflex</td>
<td>18.5</td>
<td>30.3</td>
</tr>
<tr>
<td>Heptane</td>
<td>40.4</td>
<td>66.1</td>
</tr>
<tr>
<td>ATB Precipitate</td>
<td>1.57</td>
<td>71.45</td>
</tr>
</tbody>
</table>

**Table 4.3 Component ratio of the samples and the percentage of ATB which precipitated - Time period: 3 days**

<table>
<thead>
<tr>
<th></th>
<th>Solution(1)</th>
<th>Solution(2)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Weight(g)</td>
<td>%</td>
</tr>
<tr>
<td>ATB</td>
<td>2.0</td>
<td>3.3</td>
</tr>
<tr>
<td>Paraflex</td>
<td>18.0</td>
<td>30.0</td>
</tr>
<tr>
<td>Heptane</td>
<td>40.0</td>
<td>66.7</td>
</tr>
<tr>
<td>ATB Precipitate</td>
<td>0.99</td>
<td>49.55</td>
</tr>
</tbody>
</table>
Table 4.2, with one-day of contact with Paraflex presents some 71.5% of the ATB precipitated, whereas with no Paraflex, only 15.4% of the ATB precipitated. For the three-day experiment, Table 4.3 shows that roughly the same percentage is precipitated whether Paraflex is present or not.

Table 4.4 Component ratio of the samples and the percentage of ATB which precipitated - Time period: 3 days - Duplicate experiment

<table>
<thead>
<tr>
<th></th>
<th>Solution(1)</th>
<th>Solution(2)</th>
<th>Solution(3)</th>
<th>Solution(4)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Weight(g)</td>
<td>%</td>
<td>Weight(g)</td>
<td>%</td>
</tr>
<tr>
<td>ATB</td>
<td>2.6</td>
<td>3.5</td>
<td>2.0</td>
<td>3.3</td>
</tr>
<tr>
<td>Paraflex</td>
<td>40.7</td>
<td>54.8</td>
<td>17.8</td>
<td>29.8</td>
</tr>
<tr>
<td>Heptane</td>
<td>31.0</td>
<td>41.7</td>
<td>40.0</td>
<td>66.9</td>
</tr>
<tr>
<td>ATB Precipitated</td>
<td>1.44</td>
<td>55.40</td>
<td>0.86</td>
<td>43.2</td>
</tr>
</tbody>
</table>

Table 4.5 Component ratio of the samples and the percentage of ATB which precipitated - Time period: 3 days - Duplicate experiment

<table>
<thead>
<tr>
<th></th>
<th>Solution(1)</th>
<th>Solution(2)</th>
<th>Solution(3)</th>
<th>Solution(4)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Weight(g)</td>
<td>%</td>
<td>Weight(g)</td>
<td>%</td>
</tr>
<tr>
<td>ATB</td>
<td>2.0</td>
<td>2.8</td>
<td>2.1</td>
<td>3.5</td>
</tr>
<tr>
<td>Paraflex</td>
<td>40.6</td>
<td>55.9</td>
<td>18.0</td>
<td>29.9</td>
</tr>
<tr>
<td>Heptane</td>
<td>30.0</td>
<td>41.3</td>
<td>40.1</td>
<td>66.6</td>
</tr>
<tr>
<td>ATB Precipitated</td>
<td>1.25</td>
<td>62.74</td>
<td>1.1</td>
<td>52.48</td>
</tr>
</tbody>
</table>

According to the results in Table 4.2-Table 4.5 the amount of precipitant in the presence of Paraflex in the diluent mixture is significant, while having no Paraflex in the mixture results in
less precipitation (Table 4.4, solution 4). In other words Paraflex can act as a precipitant when it is mixed with ATB.

Figure 4.2 gives us a better view of what happens when different ratios of n-Heptane and Paraflex are mixed with ATB. As the figure shows, with the percentage of n-Heptane increasing (Paraflex percentage decreasing) in the sample, less ATB is precipitated. In other words, increasing paraflex in the solution makes ATB less stable, and causes more ATB to precipitate.

![Graph](a)

![Graph](b)

**Figure 4.2** (a) Percentage of ATB which precipitated vs. % n-Heptane in the solution  
(b) Percentage of ATB which precipitated vs. % Paraflex in the solution

Figure 4.3 (a) and (b) show dried filters after filtration process.
Figure 4.3 Filters with precipitate (a) ATB Paraflex Heptane solution filtration (time period: 1 day), (b) ATB Heptane solution filtration (time period: 1 day)

4.1.3 Comparison of the effect of Paraflex and Voltesso on precipitation process in ATB

In section 4.1.2, the effect of addition of Paraflex to ATB, on the precipitation process was investigated and it was shown that addition of Paraflex to ATB increases the amount of precipitate. Similar experiments were done, this time using Voltesso in place of Paraflex. Using Voltesso would improve the heat transfer as well as circulation of ATB in the loop. To increase the accuracy of the results duplicate experiments were performed.

The results are summarized in the tables below.
### Table 4.6 Component ratio of the samples and the percentage of precipitate with respect to ATB - Time period: 3 days

<table>
<thead>
<tr>
<th></th>
<th>Solution(1)</th>
<th>Solution(2)</th>
<th>Solution(3)</th>
<th>Solution(4)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Weight(g)</td>
<td>%</td>
<td>Weight(g)</td>
<td>%</td>
</tr>
<tr>
<td>ATB</td>
<td>2.1</td>
<td>2.9</td>
<td>3.0</td>
<td>4.9</td>
</tr>
<tr>
<td>Heptane</td>
<td>30.3</td>
<td>41.7</td>
<td>40.0</td>
<td>65.6</td>
</tr>
<tr>
<td>Voltesso</td>
<td>40.3</td>
<td>55.4</td>
<td>18.0</td>
<td>29.5</td>
</tr>
<tr>
<td>ATB Precipitated</td>
<td>0.35</td>
<td>16.65</td>
<td>0.76</td>
<td>25.38</td>
</tr>
</tbody>
</table>

### Table 4.7 Component ratio of the samples and the percentage of precipitate with respect to ATB - Time period: 3 days-Duplicate experiment

<table>
<thead>
<tr>
<th></th>
<th>Solution(1)</th>
<th>Solution(2)</th>
<th>Solution(3)</th>
<th>Solution(4)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Weight(g)</td>
<td>%</td>
<td>Weight(g)</td>
<td>%</td>
</tr>
<tr>
<td>ATB</td>
<td>2.2</td>
<td>3.0</td>
<td>2.1</td>
<td>3.5</td>
</tr>
<tr>
<td>Heptane</td>
<td>30.0</td>
<td>41.1</td>
<td>40.0</td>
<td>66.1</td>
</tr>
<tr>
<td>Voltesso</td>
<td>40.8</td>
<td>55.9</td>
<td>18.4</td>
<td>30.4</td>
</tr>
<tr>
<td>ATB Precipitated</td>
<td>0.39</td>
<td>17.67</td>
<td>0.63</td>
<td>30.18</td>
</tr>
</tbody>
</table>
Results from the duplicate experiment (2) were all greater than the results from the first experiment (1). There was no apparent reason for the observed difference. Errors were larger where the amounts of precipitate were larger. The trends of both experiments were very similar. As shown in Figure 4.4, as the percentage of Voltesso was increased up to 20 %, the percentage of ATB which was precipitated increased as well. But having more than 20 % Voltesso in the mixture will resulted in less precipitation. Figure 4.5 compares the effect of using Paraflex and Voltesso as diluent for ATB on the amount of precipitate.
As presented in Figure 4.5 at low percentage of diluent (up to 20 %) in the solution the amount of precipitate is approximately the same for both Voltesso and Paraflex. But with percentage of diluent increasing, Paraflex causes more precipitation while adding Voltesso decreases the precipitate. Thus choosing Paraflex as a diluent for ATB would be reasonable, as it causes more precipitation than other choices. Table 4.8 and Table 4.9 present properties of Paraflex HT10 and ATB respectively.

A separate set of tests was carried out by Co-op student, Alex Young, using the two-component blend of Paraflex and ATB. Procedures were slightly different. In Appendix D,
Figure D. 1 and Figure D. 2 show his results, which support the present results with respect to the role of % Paraflex concentration in the blend on % ATB precipitated.

Table 4.8 Properties of Paraflex HT 10, Provided by Petro Canada Ltd.

<table>
<thead>
<tr>
<th>Properties</th>
<th>PFX</th>
</tr>
</thead>
<tbody>
<tr>
<td>SARA analysis</td>
<td></td>
</tr>
<tr>
<td>Saturates, wt%</td>
<td>99.4</td>
</tr>
<tr>
<td>Aromatics, wt%</td>
<td>0.6</td>
</tr>
<tr>
<td>Resins, wt%</td>
<td>0.0</td>
</tr>
<tr>
<td>Asphaltenes, wt%</td>
<td>0.0</td>
</tr>
<tr>
<td>Elemental analysis</td>
<td></td>
</tr>
<tr>
<td>C, wt%</td>
<td>85.66</td>
</tr>
<tr>
<td>H, wt%</td>
<td>13.73</td>
</tr>
<tr>
<td>S, wt%</td>
<td>0.0</td>
</tr>
<tr>
<td>N, wt%</td>
<td>0.0</td>
</tr>
<tr>
<td>H/C (atomic)</td>
<td>1.92</td>
</tr>
<tr>
<td>Kinematic viscosity</td>
<td></td>
</tr>
<tr>
<td>25°C (m²/s,×10⁶)</td>
<td>17.91</td>
</tr>
<tr>
<td>40°C (m²/s,×10⁶)</td>
<td>10.37</td>
</tr>
<tr>
<td>85°C (m²/s,×10⁶)</td>
<td>3.63</td>
</tr>
<tr>
<td>Molecular Weight</td>
<td>314</td>
</tr>
<tr>
<td>Specific gravity</td>
<td>0.8554</td>
</tr>
<tr>
<td>Boiling curve</td>
<td></td>
</tr>
<tr>
<td>1wt%</td>
<td>293°C</td>
</tr>
<tr>
<td>10wt%</td>
<td>324°C</td>
</tr>
<tr>
<td>30wt%</td>
<td>344°C</td>
</tr>
<tr>
<td>50wt%</td>
<td>358°C</td>
</tr>
<tr>
<td>70wt%</td>
<td>374°C</td>
</tr>
<tr>
<td>90wt%</td>
<td>400°C</td>
</tr>
<tr>
<td>99wt%</td>
<td>465°C</td>
</tr>
<tr>
<td>Properties</td>
<td>ATB</td>
</tr>
<tr>
<td>------------</td>
<td>-----</td>
</tr>
<tr>
<td>Saturates, wt%</td>
<td>21.2</td>
</tr>
<tr>
<td>Aromatics, wt%</td>
<td>45.5</td>
</tr>
<tr>
<td>Resins, wt%</td>
<td>19.8</td>
</tr>
<tr>
<td>Asphaltenes, wt%</td>
<td>13.5</td>
</tr>
<tr>
<td>C, wt%</td>
<td>82.82</td>
</tr>
<tr>
<td>H, wt%</td>
<td>10.32</td>
</tr>
<tr>
<td>S, wt%</td>
<td>4.47</td>
</tr>
<tr>
<td>N, wt%</td>
<td>0.45</td>
</tr>
<tr>
<td>H/C (atomic)</td>
<td>1.49</td>
</tr>
<tr>
<td>Ash, wt%</td>
<td>1.77</td>
</tr>
<tr>
<td>Kinematic Viscosity 25°C (m²/s)</td>
<td>0.38</td>
</tr>
<tr>
<td>85°C (m²/s)</td>
<td>0.00106</td>
</tr>
<tr>
<td>Molecular Weight</td>
<td>581</td>
</tr>
<tr>
<td>Specific gravity</td>
<td>1.0163</td>
</tr>
</tbody>
</table>

**Boiling curve**

| 2wt% | 298°C |
| 6wt% | 337°C |
| 10wt% | 365°C |
| 30wt% | 460°C |
| 50wt% | 547°C |
| 70wt% | 642°C |
| 80wt% | 701°C |
4.2 ATB fouling studies

The first runs to detect and measure fouling using Probe #1 are summarized in Table 4.10.

Table 4.10 Summary of fouling runs conditions with Probe #1

<table>
<thead>
<tr>
<th>Run</th>
<th>#3</th>
<th>#5</th>
<th>#7</th>
<th>#9</th>
<th>#11</th>
<th>#13</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fluid</td>
<td>% wt ATB</td>
<td>7.15</td>
<td>7.4</td>
<td>7.6</td>
<td>15</td>
<td>15</td>
</tr>
<tr>
<td></td>
<td>% wt PFX</td>
<td>92.85</td>
<td>92.6</td>
<td>92.4</td>
<td>85</td>
<td>85</td>
</tr>
<tr>
<td>Duration (h)</td>
<td>20</td>
<td>40</td>
<td>48</td>
<td>72</td>
<td>48</td>
<td>26</td>
</tr>
<tr>
<td>Probe Power (W)</td>
<td>117</td>
<td>197</td>
<td>223</td>
<td>247</td>
<td>370</td>
<td>412</td>
</tr>
<tr>
<td>Bulk Temperature (°C)</td>
<td>85</td>
<td>80</td>
<td>95</td>
<td>95</td>
<td>90</td>
<td>90</td>
</tr>
<tr>
<td>Probe Initial Temperature (°C)</td>
<td>240</td>
<td>380</td>
<td>415</td>
<td>455</td>
<td>570</td>
<td>640</td>
</tr>
<tr>
<td>Fluid Velocity (m/s)</td>
<td>0.5</td>
<td>0.31</td>
<td>0.6</td>
<td>0.5</td>
<td>0.5</td>
<td>0.5</td>
</tr>
<tr>
<td>$Re_b$</td>
<td>1400-1450</td>
<td>1100-1200</td>
<td>1950-2050</td>
<td>1600-1700</td>
<td>1690-1800</td>
<td>1800-1900</td>
</tr>
<tr>
<td>Probe Final Temperature (°C)</td>
<td>272</td>
<td>403</td>
<td>429</td>
<td>465</td>
<td>607</td>
<td>650</td>
</tr>
<tr>
<td>Deposit Thickness (mm)</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>Overall Heat Transfer Coefficient (W/m²K)</td>
<td>179-139</td>
<td>144-130</td>
<td>152-149</td>
<td>156-142</td>
<td>170-162</td>
<td>165-160</td>
</tr>
</tbody>
</table>

Compositions of about 7.2 to 7.6% ATB in PFX was chosen for the initial runs. Durations of runs are different and covered up to 72-h. Longer durations in Runs #7, #9, #11, #13 were used in the hope that fouling would be measurable with increase of time. Composition of the fluid was also changed in Runs #9, #11, and #13 and more ATB was used to increase asphaltene content, and consequently help fouling. However, this does have the effect of making the sample more stable. Fluid velocity also varies in some cases, however the Reynolds numbers are in one range and result is laminar flow regime. Bulk temperature varies from 80 to 95°C. Probe power was increased in different runs to give different probe surface temperatures;
however it was kept constant during each experiment. The runs were interrupted when the probe (core) temperature was increased by 15-30 degrees, when the overall heat transfer coefficient dropped by 5-10% and fouling was expected to happen. The two heat transfer coefficients reported for each run are the initial and final values, based on the core temperatures. With this probe, heat transfer coefficients are low and little heat is transferred to the fluid. Of note is the large increase in probe core temperature as the power was raised, reaching 640°C in Run #13. The probe temperature increase is due to the trapped heat in the probe. In other words, increasing the probe power to increase the surface temperature and making changes to the operating conditions such as heat flow, ATB concentration, velocity and bulk temperature did not result in fouling. Constant and changing parameters are plotted versus time for each run in Appendix E as for Run #3 in Figure 4.6.
Figure 4.6 Run #3 properties vs. time * Probe temperature on the graph is the core temperature
A possible reason behind poor heat transfer and no detection of fouling in the system is the thermal resistance within the probe. Thus the heating probe was analyzed in terms of heat transfer, and another unit, Probe #2, was designed with a different structure to improve heat transfer from the heating element to the fluid.

4.3 Probe #2 design

Probe #2 made use of an identical heating element as in Probe #1. However, instead of using a tubular casing over the heating element, extensions were attached to both ends of the heating element, such that the heating element itself was exposed to the fluid. A Macor (ceramic) rod (0.95 cm diameter and 25.4 cm long) was used to extend the upstream length of the probe. The rod was threaded so that it could be connected to the heating element via a screw that had been drilled into the bottom section of the heating element. A tube of stainless steel (SS 316) (0.95 cm outside diameter and 23.6 cm long) was used to extend the probe length downstream of the heater (Top section) via a very thin sleeve as shown in Fig. 4.7(b). In other words, Probe #2 was designed to avoid the large temperature drop from core to the surface of the probe, caused by the external tubular casing. Figure 4.8 shows Probe #2 with its connection box.
Figure 4.7 Probe #2 design (a) overall (b) details
4.4 Heat transfer analysis and comparisons of Probe #1 and #2

To analyze heat transfer in Probe #1 and #2, temperature drop from core to the surface of each probe was calculated. In both probes, the heating element (provided by Gordo Sales) is made of a ceramic core, resistance wires, a layer of magnesium oxide which is highly compacted, and the stainless steel sheath, respectively from core to the surface. Thus basically there are different layers from the point where heat is generated and measured (thermocouple is placed at the center of the heater) to the surface, which causes a temperature drop from core to the surface. In other words, wall resistance against heat transfer results in a temperature drop. There are different methods available (experimental and theoretical) to calculate the temperature drop from core to the surface.

Figure 4.9 (a) Heating element structure provided by Gordo Sales (b) Heating element
### 4.4.1 Wilson plot method

An experimental approach is to apply the Wilson plot method which is based on the separation of the overall thermal resistance into convective thermal resistances and the other conductive thermal resistances participating in the heat transfer. The Wilson plot method has been applied to numerous situations. In the present case the overall thermal resistance could be written as:

\[ R_{overall} = R_{wall} + \frac{1}{h} \]  \hspace{1cm} (4.1)

where \( R_{wall} \) represents the thermal resistance of all solid layers in the probe and is almost constant \( (C_1) \) and \( h \) is the convective film heat transfer coefficient which under chosen conditions is proportional to a power of velocity. The power would vary depending on the flow regime, typically being 0.33 for laminar flow and 0.8 for turbulent flow in annulus. Thus for experiments where only the velocity is varied and all physical properties of the fluid and probe are assumed constant, the overall thermal resistance is as follows:

\[ R_{overall} = C_1 + \frac{1}{C_2 v^n} \]  \hspace{1cm} (4.2)

On the other hand the overall thermal resistance could be obtained experimentally by measuring core temperature of the heating element, the bulk temperature of the fluid and the magnitude of the heat flux, \( Q/A_o \).

\[ R_{overall} = \frac{A_o (T_{core} - T_{bulk})}{Q} \]  \hspace{1cm} (4.3)

Measuring \( Q \), core, and bulk temperatures at different flow velocities, the overall heat transfer resistance could be plotted versus \( \frac{1}{v^n} \) (where \( n \) is a positive value between 0 and 1 that yields the best linear fit, in this case 0.62) The intercept yields the wall resistance \( C_1 \) and
according to the heat transfer equation from the core to the surface the temperature drop in this region can be expressed as a function of input power.

\[
(T_{\text{core}} - T_{\text{surface}}) = \frac{R_{\text{wall}}Q}{A_o}
\] (4.4)

This method was used for both probes for conditions given in Table 4.11. The results are compared, although the experiment conditions are a little different.

<table>
<thead>
<tr>
<th>Table 4.11 Wilson plot method run properties for Probe #1 and #2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fluid</td>
</tr>
<tr>
<td>-------</td>
</tr>
<tr>
<td>Tank Pressure (kPa, abs)</td>
</tr>
<tr>
<td>Tank temperature (°C)</td>
</tr>
<tr>
<td>Heating probe power (Watts)</td>
</tr>
<tr>
<td>Flow velocity range (m/s)</td>
</tr>
<tr>
<td>Re_0</td>
</tr>
<tr>
<td>Heated Area (m²)</td>
</tr>
</tbody>
</table>

![Figure 4.10 Wilson plot method for clean heat transfer coefficients at constant heat flux for Probe #1 and Probe #2](image)

\[
y = 0.8382x + 5.304 \\
R^2 = 0.9332
\]

\[
y = 0.8315x + 0.4972 \\
R^2 = 0.9939
\]
The heat transfer coefficient for Probe #2 (400-800 \( W/m^2.K \)) is almost four times larger than for Probe #1 (130-150 \( W/m^2.K \)), which means that the heat is being transferred better using Probe #2. The intercepts for Probe #1 and #2 are \( 5.304 \times 10^{-3} \) and \( 0.4972 \times 10^{-3} \) (\( m^2/kW \)) respectively. Thus using equation 4.4 and reading heat transfer area from Table 4.11, the temperature drop in the probe for each would be as follows:

Probe #1
\[
(T_{core} - T_{surface}) = 1.18 \ Q \tag{4.5}
\]

Probe #2
\[
(T_{core} - T_{surface}) = 0.15 \ Q \tag{4.6}
\]

Based on these two equations at a specific heat flow (power) the radial temperature drop through Probe #2 is much lower (factor of 7.8) than for Probe #1.

### 4.4.2 Boiling water method

If a thermocouple could be connected to the surface of the probe, the surface temperature could be measured directly. But as it was not feasible to make such changes to the fouling probe, a thermocouple is attached to the probe surface using a thin wire, and the probe was immersed in water. It is recognized that this attached thermocouple would affect the flow over the probe. Another thermocouple was used to record the bulk temperature of water near the probe. The power was gradually increased to see bubbles on the surface and the core temperature of the probe as well as surface and bulk temperatures were recorded to get the temperature drop from core to the surface. However, since water is not flowing except by natural convection, a weaker heat transfer and lower heat transfer coefficient is expected. In other words heat will be trapped in the probe and consequently core temperature keeps rising and the temperature difference from core to the surface becomes large. Axial heat flow would also become important. This
experiment was done using Probe #1 only, and the results are summarized in Figure 4.11

Figure 4.11 Water experiment with Probe #1.
At 100°C probe surface temperature, where bubbles were seen on the surface of the probe, the core temperature was 250°C. This experiment also suggests that at power of 220 watts temperature drops from 370 °C to 120 °C, which is a drop of 67% from core to the surface.

Figure 4.12 \((T_{\text{core}} - T_s)\) from water experiment and expected temperature drop from Wilson plot method versus time for Probe #1
Figure 4.12 compares the expected temperature drop from the Wilson plot method and the one from the boiling water experiment. At power of 220 watts the expected temperature drop by Wilson plot method is 70%, which is comparable with the experimental method.

4.4.3 Theoretical method

A third way to calculate the temperature drop from core to surface of the heating element is to use the theoretical radial heat conduction equations. As the thickness of each layer and the thermal conductivity of each material are known, the temperature drop at each step can be calculated to get the temperature at the surface, for a given heat flow. Axial heat conduction is ignored.

In Probe #1, the heating element is placed inside of a stainless steel tube (OD 0.0127 m, ID 0.0085 m) casing much longer than itself, such that the heating element is located in an appropriate axial position where the flow could be established before being exposed to heat, and the fluid exit at the top of the test section would be satisfying for downstream of the heated section that it would not interfere with the flow. The casing tube was drilled out to a diameter of 0.0105 m as discussed in section 3.1.4, and the heater with outer diameter of 0.0095 m placed inside it, and shrunk fit to be tight. Thus there could be a 0.001 m free space of air between the heater and the casing tube (0.0005 m on each side). However due to the roughness that is not uniform inside the tube, the exact thickness of this air layer is not clear, and as the heater was shrunk fitted inside the tube, it could be claimed that heater and its casing are in contact at some point. In the assumed air gap (zone 5 in Figure 4.13) heat would be transferred through natural convection if the gap was sufficiently wide for this type of heat transfer to occur. Thus an effective conductivity, $k_e$, is used instead of the molecular thermal conductivity, $k$, in the
conduction equation (Equation 4.10). Figure 4.13 shows the layers considered, and structure in Probe #1.

Probe #2 was designed differently. Figure 4.14 shows the layers considered and structure of Probe #2.

The general equation of heat conduction in cylindrical coordinates can be written as:

$$\frac{1}{r} \frac{\partial}{\partial r} \left( kr \frac{\partial T}{\partial r} \right) + \frac{1}{r^2} \frac{\partial}{\partial \phi} \left( k \frac{\partial T}{\partial \phi} \right) + \frac{\partial}{\partial z} \left( k \frac{\partial T}{\partial z} \right) + q(r, \phi, z) = \rho C_p \frac{\partial T}{\partial t}$$  \hspace{1cm} (4.7)

The radial heat transfer conduction equation through the layers of the probe, for steady-state, clean heat transfer condition where there is no temperature change in the axial direction (z coordinate) and no circumferential change around the probe (ϕ coordinate) from Equation 4.7 is as follows:

$$\frac{k \partial T}{r \frac{\partial}{\partial r}} + k \frac{\partial^2 T}{\partial r^2} + q(r) = 0$$  \hspace{1cm} (4.8)

where k is the thermal conductivity and q (r) is constant because of the uniform energy generation. The equation could be solved analytically for each layer with its own boundary condition for both probes. Then by substituting the real values of energy generation (Probe power) and the core temperature given by the thermocouple, the corresponding surface temperature can be determined.

For Probe #1, considering Figure 4.13, we will have:

$$T_6(r) = T_6 - \frac{Q}{2\pi L k_6} \ln \left( \frac{r}{R_6} \right)$$  \hspace{1cm} (4.9)

where \(T_6\) and \(R_6\) are temperature and radius respectively at outer edge of the sixth layer. \(T_6(r)\) gives the temperature distribution in zone number 6, and \(k_6\) is the thermal conductivity in this zone.
\[ T_5(r) = T_6(R_5) - \frac{Q}{2\Pi L k_e} \ln \left( \frac{r}{R_5} \right) \] (4.10)

\[ T_4(r) = T_5(R_4) - \frac{Q}{2\Pi L k_4} \ln \left( \frac{r}{R_4} \right) \] (4.11)

\[ T_3(r) = T_4(R_3) - \frac{Q}{2\Pi L k_3} \ln \left( \frac{r}{R_3} \right) \] (4.12)

\[ T_2(r) = T_3(R_2) + \frac{Q}{4\Pi L (R_2^2 - R_1^2) k_2} (R_2^2 - r^2) + \frac{Q}{2\Pi L (R_2^2 - R_1^2) k_2} R_1 \ln \frac{r}{R_2} \] (4.13)

\[ T_1(r) = T_2(R_1) \] (4.14)

In other words, equations 4.9 to 4.14 give the temperature distribution in each layer for Probe #1.

While for Probe #2, Equation 4.9 and 4.10 are not applied, considering Figure 4.14.
Figure 4.13 Schematic temperature profile in different radial zones of Probe #1
(see Table 4.12 for dimensions)

Figure 4.14 Schematic temperature profile in different radial zone of Probe #2
(see Table 4.12 for dimensions)
Probe #1:

Calculations suggest that for a power of 412 W (Run #13) which results in a measured core temperature of 640°C, the predicted surface temperature will be 337°C. The temperature drops by 47%. Using this calculation \((T_{\text{core}} - T_s) = 303°C\), and thermal resistance (R) is 3.35\((m^2K/kW)\). Table 4.12 shows specifications and the temperature at the boundary of each zone. The very large thermal resistance of the wall of this probe suggests it will be poor for detecting small extents of fouling.

<table>
<thead>
<tr>
<th>Zone</th>
<th>Material</th>
<th>Thickness(mm)</th>
<th>R(m)</th>
<th>k(W/(m.K))</th>
<th>T(°C) at the outer edge of zone</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Ceramic</td>
<td>2.65</td>
<td>0.00265</td>
<td>35</td>
<td>640</td>
</tr>
<tr>
<td>2</td>
<td>Stainless Steel</td>
<td>0.3</td>
<td>0.00295</td>
<td>18.5</td>
<td>638</td>
</tr>
<tr>
<td>3</td>
<td>Magnesium Oxide</td>
<td>0.9</td>
<td>0.00385</td>
<td>15</td>
<td>627</td>
</tr>
<tr>
<td>4</td>
<td>Stainless Steel</td>
<td>0.9</td>
<td>0.00475</td>
<td>18.5</td>
<td>620</td>
</tr>
<tr>
<td>5</td>
<td>air</td>
<td>0.5</td>
<td>0.00525</td>
<td>0.21</td>
<td>343</td>
</tr>
<tr>
<td>6</td>
<td>Stainless Steel</td>
<td>1.1</td>
<td>0.00635</td>
<td>18.5</td>
<td>337</td>
</tr>
</tbody>
</table>

Similar calculations were done assuming no air gap (zero thickness of air in Table 4.12 and 1.6 mm. thickness of stainless steel instead of 1.1 in the same table). These yielded a temperature of 611°C for the outer edge, a temperature drop of 29°C (4.5%), and a thermal resistance equal to 0.31\((m^2K/kW)\).

Probe #2:

A similar calculation was done for the second probe. The difference in the structure and lack of the air gap in this case, results in a lower temperature drop in theory. For Probe #2, there are only four zones (Four top rows of Table 4.12). And for the probe power of 308 W and
measured core temperature of 220 °C the surface temperature drops to 205°C, which means 15°C of temperature drop (6%) is expected in Probe #2, and the thermal resistance (R) is 0.22\(\left(\frac{m^2 K}{W}\right)\).

Table 4.13 Different zones in Probe #2 with corresponding material, thickness, radial distance from the core, thermal conductivity, and temperature on the outer edge.

<table>
<thead>
<tr>
<th>Zone</th>
<th>Material</th>
<th>Thickness(mm)</th>
<th>R(m)</th>
<th>k(W/(m.K))</th>
<th>T(°C) at the outer edge of zone</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Ceramic</td>
<td>2.65</td>
<td>0.00265</td>
<td>35</td>
<td>220</td>
</tr>
<tr>
<td>2</td>
<td>Stainless Steel</td>
<td>0.3</td>
<td>0.00295</td>
<td>18.5</td>
<td>218</td>
</tr>
<tr>
<td>3</td>
<td>Magnesium Oxide</td>
<td>0.9</td>
<td>0.00385</td>
<td>15</td>
<td>210</td>
</tr>
<tr>
<td>4</td>
<td>Stainless Steel</td>
<td>0.9</td>
<td>0.00475</td>
<td>18.5</td>
<td>205</td>
</tr>
</tbody>
</table>

From these theoretical calculations, the temperature drop in Probe #2 is much less than in Probe #1, which makes sense since in Probe #2 there is no air gap and no extra layer of a casing. Besides, from experience the surface temperature needed to cause measurable fouling for a mixture of 7.5 % ATB in Paraflex is about 315 °C which is not met by Probe #1 even at its highest possible core temperature.

For Probe #2 theoretical temperature drop is compared with the temperature drop calculated from Wilson plot method at different probe heating levels. Figure 4.15 shows that for lower power to the probe, both methods give the similar temperature drops, and as the probe power increases they give different results. The theoretical calculation here, ignores the convective cooling of the probe surface by the flowing fluid, which could be incorporated using equations for convective heat transfer coefficients in the annular geometry. The Wilson-plot also has uncertainties as it relies on extrapolations, and has been applied here in the transition region.
4.4.4 Conclusion - Heat transfer analysis of Probe #1 and #2

Both experimental and theoretical methods confirm that in Probe #1 a very large temperature drop occurs compared to that of Probe #2 due to the different construction. In terms of consistency of the methods, the results from Wilson plot and water experiment are compatible as discussed in section 4.4.2. The theoretical method and Wilson plot are compatible at low input powers. The most reliable method seems to be the theoretical one as it is based on heat transfer equations that are applicable at any conditions, while the Wilson Plot method is dependent on the relationship between velocity and convective heat transfer coefficient, which varies depending on the flow regime.

Figure 4.15- Temperature drop from core to the surface vs. probe power for Probe #2
(Wilson plot data are in Figure 4.10)
4.5 ATB fouling studies (using Probe#2)

Other than clean heat transfer runs conducted using Probe #2 to get the Wilson plot data (Figure 4.10), fouling runs were also conducted with this probe. Fouling run conditions are summarized below:

<table>
<thead>
<tr>
<th>Table 4.14 Conditions of Run #17</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Fluid</strong></td>
</tr>
<tr>
<td><strong>Duration (h)</strong></td>
</tr>
<tr>
<td><strong>Probe Power(W)</strong></td>
</tr>
<tr>
<td><strong>Bulk Temperature(°C)</strong></td>
</tr>
<tr>
<td><strong>Probe Initial Temperature (°C)</strong></td>
</tr>
<tr>
<td><strong>Fluid Velocity (m/s)</strong></td>
</tr>
<tr>
<td><strong>Re</strong></td>
</tr>
<tr>
<td><strong>Probe Final Temperature (°C)</strong></td>
</tr>
<tr>
<td><strong>Deposit Thickness (mm)</strong></td>
</tr>
<tr>
<td><strong>Overall Heat transfer coefficient (W/m²K) (Based on T_core)</strong></td>
</tr>
</tbody>
</table>

Comparing the results with Table 4.10, the heat transfer coefficient is much higher (630-700 W/m²K) when using Probe #2 than for Probe #1 (130-179 W/m²K, Table 4.10). As well, to get the same probe surface temperature, higher applied power is needed for Probe #2, which means that heat is dissipating into the fluid in this case. With the initial conditions mentioned in Table 4.14, after 2 hours of initiation, in less than 20 hours the probe temperature reached the maximum set point (350°C) and the system shut off automatically. Changes of the parameters over time after initiation period are plotted in Figure 4.16.
Figure 4.16 Run #17 properties vs. time

* Probe temperature on the graph is the core temperature
Fouling resistance \((R_f)\) keeps increasing, with the overall heat transfer coefficient decreasing, as fouling occurs. The heat transfer coefficient drops by 10% due to fouling and the probe temperature increases by 11% as the fouling layer acts as a thermal resistance. The average fouling rate equals \(0.0026\left(\frac{m^2K}{kWh}\right)\). Figure 4.17 shows the heated section of the probe, before and after fouling, as well as after getting cleaned.

![Heated section of the probe, (a) before fouling, (b) after fouling, and (c) after getting cleaned](image)

Figure 4.17 Heated section of the probe, (a) before fouling, (b) after fouling, and (c) after getting cleaned

A further experiment was needed to confirm the results. However due to the leakage of the oil into the heater, it was found to have been shorted out. Figure 4.18 shows oil leakage into the heater, where the white MgO of the original probe appears dark brown. The leakage is due to the sleeve used to connect the top piece of tubing to the heating element. This piece had not been welded at a high temperature, as the thermocouple and power wire might have been damaged.
Figure 4.18 Oil leakage into the heater

To build another probe, it is suggested to use an insulator around the wires, to make sure they will not be damaged, and weld the sleeve at a higher temperature to provide a seal and avoid any leakage of oil into the heater.
4.6 Calculation of wall shear stress for prior annular fouling studies at UBC

The UBC annular fouling loop has been used over many years for various experiments on fouling of different oils. How fouling is affected by temperature changes, velocity changes, and oil composition and stability are some of the prior investigations done using the fouling loop. However, the importance of inner wall shear stress and its effects on fouling rate has not been explored. In this work, the role and effect of inner wall shear stress on fouling rate is investigated and compared with the correlations available in the literature, using the data of previous studies. In other words, from the data recorded by other researchers using essentially the same heat transfer loop, further calculations were done to determine the inner wall shear stress and its effect on the fouling rate is discussed, and compared with the correlations available in the literature. The heaters used were the “HTRI type Probe” which are no longer available.

Three different theses are studied: Asomaning’s PhD thesis (Asomaning, 1997), Srinivasan’s Master’s thesis(Srinivasan,2008) , and Saleh’s PhD thesis(Saleh, 2005). Many elements vary in these theses from one to another, such as oils used, presence of diluent, bulk temperature, heating surface temperature, flow velocity and consequently regime, etc. However, in all cases the fouling rate is measured, and also the effect of velocity on fouling rate. Viscosity and density data are also presented in each case. Thus the experiment is repeated at various velocities, at fixed surface temperatures. In no case was pressure drop measured, or shear stress values reported. Considering that wall shear stress could be calculated from velocity, the fouling rate is therefore available at different inner wall shear stresses. A summary of each work is presented below:
4.6.1 Asomaning’s Thesis

Asomaning’s work (1997) included a set of experiments to investigate the effect of bulk velocity on fouling rate. The fluid was a mixture of Heavy oil (10% wt) and Fuel oil (90% wt) at bulk temperature of 85°C. Compositions and properties of the two oils were given. The initial probe surface temperature was 220°C, the experiments were done at different fluid bulk velocities (ranged from 0.65 to 0.95 m/s), and the fouling resistance was measured up to 35 hours. Although velocity range was not so wide, the experiments yielded five data points. It should be mentioned that $d_{out} - d_{in}$ was used as equivalent diameter, and bulk temperature was used in the Reynolds number calculation, which resulted in a range of Reynolds number between 1600 and 2310 (Laminar, transitional region). Based on the assumed definition for equivalent diameter and the temperature at which Reynolds number is calculated this value can change.

4.6.2 Srinivasan’s thesis

Srinivasan, as part of his Master’s thesis (2008) made a detailed fouling study on Light Sour crude oil, including the effect of velocity on fouling rate. Experiments were carried out in a different fouling unit than Asomaning (1997) and the annular section and conditions of the experiments were quite different. The bulk temperature was much higher ($T_b = 275.5°C$) than in Asomaning’s work, the initial probe surface temperature was also higher ($T_{s0} = 375°C$). No diluent was used as the bulk temperature was high enough for the oil to flow. The heater rod was the same, but the inside diameter of the outer shell was smaller, which gave a different equivalent diameter and consequently a different cross-sectional area for flow. Due to the limitation of the pump the highest velocity was 0.75 m/s. However, since the temperature was high, the range of Reynolds number was higher and wider (1160-5764). The fouling rate was
calculated for up to 48 hours. Fluid properties (viscosity and density) were calculated at film temperature. Thus Reynolds number was calculated at film temperature.

4.6.3 Saleh’s thesis

Saleh (2005) did experimental work at UBC, while a graduate student at the University of New South Wales. His work included some fouling experiments focusing on the effect of different operating parameters such as velocity, pressure, surface and bulk temperature on thermal fouling of Gippsland crude oil and Bach Ho crude oil. The tests were done at moderate temperatures compared to those of Asomaning (1997) and Srinivasan (2008) both for bulk and surface, 80°C and 250°C respectively. Pressure range was 445-800 (kPa, abs), and velocity range for Gippsland and Bach Ho oil were 0.25-0.45 and 0.25-0.75 m/s respectively. This velocity range for Gippsland oil resulted in a Reynolds number from 2071 to 3148 on bulk temperature basis, and for Bach Ho oil this value was from 1584 to 4700. The duration of each run was 90 hours.

4.6.4 Wall Shear Stress Calculation

The sets of experiments mentioned above, yield fouling rates of various oils at different velocities. The widest velocity ranges produces the widest shear stress range. However, finding an appropriate equation to get the inner wall shear stress in each case was an issue. Meter and Bird (1964) define the inner wall shear stress in an annulus for any flow regime as follows:

$$\tau_in = \frac{r_{out} \Delta p}{2L} \left(\frac{a^2 - \lambda^2}{a}\right)$$

(4.15)

where \(a\) is the radius ratio and \(\lambda\) is the dimensionless radial distance of the maximum velocity, as mentioned in section 2.2.1.
They also use the following equation for the pressure drop for fully developed flow under isothermal condition:

\[
\Delta p = \frac{8\mu T_b L v_{av}}{r_{out}^2 \left[1 + a^2 - \frac{1 - a^2}{\ln \left(\frac{1}{a}\right)}\right]} \quad (4.16)
\]

However, in case of convective heat transfer, pressure drop should be calculated differently. Thomas (1999) discusses the forced convection flow in annuli.

### 4.6.4.1 Pressure Drop Recommendation of Thomas (1999)

One approach to categorize convective heat transfer according to the geometry of heat transfer surface is to consider the internal flow in circular tubes, annuli, and noncircular tubular passages with uniform cross-sectional area. In practical analysis of forced convection heat transfer, coefficients such as friction factor and values of pressure drop and heat transfer rate are needed. A correlation introduced by Langhaar (1942) to characterize the pressure drop is as follows:

\[
\Delta p = \frac{2L}{D_e} f_{app} \rho v_{av}^2 \quad (4.17)
\]

Where \(f_{app}\) is the apparent friction factor. In modern heat exchanger design another form of this relationship is used, which allows for entrance losses.

\[
\Delta p = \frac{\rho v_{av}^2}{2} (K_c + \frac{4L}{D_e f_f}) \quad (4.18)
\]

Where \(f\) is the fanning friction factor and \(K_c\) is the entrance-loss coefficient for fully developed flow. Hydrodynamic and thermal fully developed flow can occur for

\[
\frac{L}{D_e} \geq 0.05 \text{ } Re \quad \text{HFD}
\]
\[ \frac{L}{D_e} \geq 0.05 \text{Re Pr} \quad \text{TFD} \]

For laminar flow f could be found in Table (Thomas, 1999, p.464) as dependent on radius ratio.

Table 4.15 HFD and TFD laminar flow coefficient for calculation of f

<table>
<thead>
<tr>
<th>Geometry</th>
<th>(C_1 = f\cdot \text{Re})</th>
</tr>
</thead>
<tbody>
<tr>
<td>Square tube</td>
<td>14.2</td>
</tr>
<tr>
<td>Circular tube</td>
<td>16</td>
</tr>
<tr>
<td>Infinite parallel plates</td>
<td>24</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Di/Do</th>
<th>0</th>
<th>0.05</th>
<th>0.1</th>
<th>0.2</th>
<th>0.4</th>
<th>0.6</th>
<th>0.8</th>
<th>1</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>16</td>
<td>21.57</td>
<td>22.34</td>
<td>23.09</td>
<td>23.68</td>
<td>23.9</td>
<td>23.98</td>
<td>24</td>
</tr>
</tbody>
</table>

Kays and London (1984) provided the following Figure (Thomas, 1999, p. 467) for \(K_c\), the entrance-loss coefficient as a function of contraction ratio, which is cross-sectional area divided by frontal area.
Having $K_c$ and $f$, we can use Equation 4.18 to calculate the pressure drop.

4.6.4.2 Heat Transfer and Pressure Drop Equations of Taborek (1997)

Taborek (1997) discusses the double-pipe heat exchanger with plain tubes in terms of heat transfer and flow on annulus side, for laminar, transitional, and turbulent flow. In his case the heat is transferred between two liquids; instead of having a constant heat flux inside the inner tube, a liquid flows. To determine the pressure drop, he uses the tubular relations in which the equivalent diameter of the annulus replaces the tube inside diameter. He also considers the losses due to entrance and exit of the fluid and calls this resistance factor $K$. There are two nozzles on entrance and exit side on a U-tube annulus and $K$ is usually 1.5 in this case. Equation 4.19 shows how he calculates the pressure drop.
\[ \Delta p = \left[ f_D \left( \frac{\mu_b}{\mu_w} \right)^{-0.14} \left( \frac{L}{D_e} \right) + K_c \right] \left[ \frac{\rho v_{bulk}^2}{2} \right] \] (4.19)

The viscosity term in this equation is for non-isothermal situation. L is the total length of the tube in the double pipe exchangers. \( v_{bulk} \) is the bulk velocity inside the annulus. \( f \) is the Darcy friction factor which is different for different flow regimes.

- Laminar flow \( f = \frac{64}{Re} \)  
- Turbulent flow \( f = [0.79 \ln(Re) - 1.64]^2 \)

For transition flow the larger of the two values is recommended.

Shah’s equation (Shah, 1975, p. 89) for the pressure drop calculation is almost the same as Taborek’s, except that he uses the Fanning friction factor and he does not consider the viscosity effect as follows:

\[ \Delta p = \left[ f_D \left( \frac{L}{D_e} \right) + K_c \right] \left[ \frac{\rho v_b^2}{2} \right] \] (4.21)

In other words Taborek’s equation is a modified version of Shah’s and is more appropriate in the present case, as pressure drop is evaluated during heat transfer.
4.6.5 A comparison between shear stress calculated using equations of Taborek and Langhaar

There are two main differences between Langhaar’s and Taborek’s methods in pressure drop calculation. First is the viscosity effect, which is included in Taborek’s equation because of heat transfer and not in Langhaar’s isothermal one. The second difference is the way friction factor is calculated for each correlation. However, friction factors are almost the same as calculations show. Thus the differences in the shear stress value are due to the viscosity effect. Using both Langhaar’s and Taborek’s equations for pressure drop, inner wall shear stress is calculated for Bach Ho oil and a mixture of 10% Heavy oil in fuel oil which are used in Saleh’s and Asomaning’s works respectively. Bach Ho oil is the less sensitive oil to temperature in terms of viscosity among the fluids used in the theses that were studied, while mixture of heavy oil and fuel oil has the greatest change in viscosity with temperature increase. Figure 4.20 shows the results of this calculation.
Considering that these correlations are suggested for laminar flow, it is obvious that as the flow moves to transitional and turbulent regime, they are less reliable, and for laminar flow depending on the sensitivity of the fluid viscosity on temperature, the viscosity changes affect them differently. In other words, the results of two correlations are closer for low Reynolds number, and for a fluid the viscosity of which does not change significantly with temperature. As is shown in Figure 4.20 for the mixture of heavy oil and fuel oil there is a greater difference between inner wall shear stress calculated from Langhaar’s equation and Taborek’s equation than for Bach Ho oil where both equations give the same shear stress.
As a general correlation, Taborek’s equation seems to be more appropriate, as viscosity changes could play a significant effect on shear stress.

Other than that, Reynolds number could be calculated both at film and bulk temperature. Film Reynolds number represents the Reynolds number near the hot surface which determines the flow regime close to the surface. In other words, to see the effect of temperature difference between bulk and surface on the flow regime, one can compare bulk and film Reynolds numbers. Viscosity of the fluid is the most sensitive property to the temperature. The equations provided in the theses to calculate the fluid viscosity as a function of temperature can be found in the Appendix A.

Figure 4.21 and Figure 4.22 show the fouling rate versus bulk and film Reynolds number respectively. Similar calculation was done for Run #17, which yields to a single data point in Figure 4.21. The result of this calculation is quite compatible with another single data point which was calculated from Hong E’s work (2005) of the same composition. Her work included studying composition effect on fouling at a constant velocity (0.75(m/s)).
Figure 4.21 Fouling rate versus film Reynolds number *(10% ATB in PFX taken from Hong E’s thesis (2005))*
As shown in the Figure 4.21 and Figure 4.22, fouling rate decreases sharply with Reynolds number (film and bulk). For film Reynolds number all the points are shifted to the right which is due to the decrease in viscosity with increasing temperature. For 10% of Heavy oil in fuel oil where the viscosity is significantly affected by temperature increase, even the flow regime changes near the hot surface. However, Asomaning provides a graph to show how viscosity of a mixture of 10% heavy oil in fuel oil changes with temperature between 30-100°C. Thus extrapolation to the temperature of 150°C, which is the film temperature, yields a very
small value for the viscosity which might not be quite reliable. Fouling rate among the various studies are better calculated with the bulk Reynolds number, which is somewhat surprising.

Inner wall shear stress is calculated using equations 4.15, 4.19 and 4.20 for the theses that have been reviewed. Considering that different fluids with different physical and chemical properties are used in the experiments, the initial fouling rates are different and are even in different ranges, as well as inner wall shear stress. As shown in Figure 4.23, a family of curves results. The highest shear stresses arise in the heavy oil blend, which has the highest viscosity (viscosity of each oil at its bulk temperature is listed in Table 4.17). In all cases the fouling rate decreases with increasing shear stress, which supports the idea that the attachment to the wall dominates over the mass transfer of the precursors from the bulk to the surface. However, the precursors will not be the same in each study, and the adhesion process is different in each.

<table>
<thead>
<tr>
<th>Fluid</th>
<th>Bulk Temperature(°C)</th>
<th>Viscosity (cSt)</th>
</tr>
</thead>
<tbody>
<tr>
<td>10wt% Heavy oil in fuel oil</td>
<td>85</td>
<td>5.95</td>
</tr>
<tr>
<td>Light sour blend oil</td>
<td>265</td>
<td>0.77</td>
</tr>
<tr>
<td>Bach Ho oil</td>
<td>80</td>
<td>2.32</td>
</tr>
<tr>
<td>Gippsland oil</td>
<td>80</td>
<td>2.02</td>
</tr>
<tr>
<td>10% ATB in PFX</td>
<td>85</td>
<td>5.04</td>
</tr>
<tr>
<td>7.5% ATB in PFX</td>
<td>85</td>
<td>5.03</td>
</tr>
</tbody>
</table>
Epstein (1994) developed a mathematical model to simulate the initial fouling rate due to the polymerization of styrene from kerosene. Based on this model, initial fouling rate is governed by mass transfer of a precursor from the bulk to the surface as well as chemical attachment to the surface. The model has been tested for different cases of chemical reaction fouling, which is the same category of fouling in the theses that have been reviewed in this work, and also for precipitation fouling of CaSO$_4$ in water (Fahiminia et al., 2007). Yeap et al. (2004, 2006) has quoted this model in its first order for crude oil refinery fouling as follows:

$$\frac{dR_f}{dt} = \frac{C_b}{k_m} + \frac{k'_2 \tau_w e^{E/RT_s}}{\mu}$$  \hspace{1cm} (4.22)
The first term in the denominator represents the mass transfer role, and the second term refers to the attachment to the wall.

In cases where the mass transfer becomes negligible compared to the attachment, equation 4.22 simplifies to

\[
\frac{dR_f}{dt} = \frac{k'_3 \mu e^{-E/RT_s}}{\tau_w} \tag{4.23}
\]

Considering equation 4.23, fouling rate divided by viscosity is plotted versus the inverse of inner wall shear stress for three different oil mixtures in Figure 4.24.

![Figure 4.24 Fouling rate/viscosity versus 1/inner wall shear stress](image-url)

**Figure 4.24 Fouling rate/viscosity versus 1/inner wall shear stress**
The data from each study results in a separate straight line, giving some support to the model. It could be claimed that for Bach Ho oil, which is best fitted to a straight line, the attachment process is absolutely dominant on the mass transfer process.
5.1 Concluding remarks

This thesis involved fouling due to precipitation of asphaltene from a blend of heavy oil fraction (ATB) and a diluent (Paraflex). Diluents were needed for the experiments due to the high viscosity of ATB. PFX and Voltoesso were tested as diluents to investigate their effect on asphaltene precipitation from ATB.

- Vacuum filtration after one and three days shows that the time period affects the asphaltene precipitation in a mixture. In this case, with the time passing more ATB is dissolved in the diluent and the precipitate weight and percentage decreases.
- The filtration tests also show that the more aliphatic PFX is a better diluent for fouling studies, as more ATB precipitates in PFX compared to Voltoesso.
- The percentage of the diluent in the mixture also affects the precipitation process. Increasing the Voltoesso portion in the mixture to more than 20 % decreases the percentage precipitation of ABT.

In preparation for fouling studies an existing flow loop was modified by the addition of a new pump, data acquisition system and annular fouling probe. Runs with Probe #1 were done for different time periods, heat flows, velocities, and ATB concentrations, but no fouling was evident.

- As probe power was raised, excessive core temperature (up to 640°C) indicated poor heat transfer to the fluid.
- Increasing % of ATB in the mixture, which would increase asphaltene concentration did not result in fouling.
- The core temperature rise with time was not a sign that fouling was occurring.
• Subsequent theoretical heat transfer calculations, as well as a Wilson-plot probe calibration indicated that the external tubular casing of Probe #1 led to large thermal resistance within the probe and a corresponding large temperature difference between the probe core and the surface, as a thin layer of air between heating element and its casing tube acts as additional thermal resistance.

• Probe #2 with its new design that eliminated the tubular casing worked much better in terms of heat transfer to the fluid. Both Wilson-plot calibration and theoretical heat conduction calculations show that the internal probe thermal resistance was reduced by about 85% from Probe #1.

A fouling run for Probe #2 was done over 67 hours on a mixture of 7.56% ATB in PFX. Power was increased to 578 (W) after the first 24 hours, and fouling occurred on the probe surface.

• For Probe #2 an 80% higher clean heat transfer coefficient was seen compared to Probe #1.

• The fouling initiation period for the mixture at the power of 578 (W) was 24 hours, following which the fouling resistance rose gradually with time.

• The design of Probe #2 is therefore recommended for future annular fouling studies.

Several prior UBC theses with focus on fouling of a heavy-fuel oil blend and different crude oils such as Bach Ho, Gippsland, and Light Sour Blend were examined. Wall shear stresses in these experiments were calculated, knowing velocity and oil properties. The effect of the inner wall shear stress and other flow parameters on fouling can be summarized as follows:

• Different oils, depending on their physical properties and chemical specifications, show different fouling rates that fall over a significant, wide range.
• Film Reynolds number, depending on the sensitivity of the fluid viscosity to temperature as well as the difference between film and bulk temperature, could be sufficiently different from the bulk Reynolds number so that it changes the flow regime from laminar to turbulent. The bulk Reynolds number could correlate fouling rates from all oils.

• Inner wall shear stress calculation from pressure drop also shows that the inner wall shear stress for a specific geometry changes significantly depending on the properties of the fluid, specially, viscosity.

• Apart from chemistry of the fluid, fouling rate decreases as the inner wall shear stress increases for all oils studies. This means that the attachment to the surface dominates over mass transfer of the foulant from the bulk to the surface.

• The intensity of the increase in fouling rate with inner wall shear stress decreasing is dependent on the fluid. To be more specific, for Bach Ho oil which is best fitted to the model that was proposed in case of attachment dominance, it could be claimed that the attachment to the wall is more effective than the mass transfer from bulk to the surface, comparing to Gippsland oil and 10% of heavy oil in fuel oil.

5.2 Recommendations for future work

• A new heating probe with a similar structure as Probe #2 should be built and tested, except that the sleeve that connects the top annulus to the heating probe should be welded well enough so that no oil leaks inside the heating elements at high pressures.

• Make some changes to the UBC fouling loop to provide higher bulk temperature as it is more likely for fouling to happen at higher bulk temperatures.

• A better pump with higher power may be helpful to get a wider range for bulk velocity.
• Modifying the system with a thermocouple that can measure the surface temperature of
  the heating probe with no influence on the flow will increase the accuracy of calculations.
• With these improvements, detailed study of wall shear stress can be done in annular flow.
References


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Wiehe IA. Tutorial on the phase behavior of asphaltenes and heavy oils. American Institute of Chemical Engineers; 1999.


Appendix A Properties of material used

Kinematic Viscosity (cSt)

Kinematic viscosities for ATB and Paraflex were calculated from Geniesse & Delbridge equation.

\[
\log(\log(\nu + 0.8)) = A + B \log(T) \quad \text{(A.1)}
\]

\(T\) in K

ATB \quad A = 9.0264, B = -3.34584

PFX \quad A = 9.7939, B = -3.7518

Asomaning (1999) provides the kinematic viscosity of 10% Heavy oil in Fuel oil between 30-100 (°C) in the Figure A.1:

![Figure A.1 Kinematic viscosity of 10% Heavy oil in Fuel oil versus temperature](image)

\[
y = 62.247e^{0.03x} \quad R^2 = 0.9794
\]
Fitting data to an exponential equation, extending the curve will give the kinematic viscosity of
the mixture at film temperature, however this long extrapolation is not reliable.

**Dynamic Viscosity (Pa.s) (Saleh, 2005)**

Bach Ho oil \[ \mu = \ln\left(\frac{1}{T(\degree C)} + 39.43\right) + 8.4980 - 2.1390 \] (A.2)

Gippsland \[ \mu = \ln\left(\frac{1}{T(\degree C)} + 42.98\right) + 8.4941 - 2.1388 \] (A.3)

**Density (kg/m³)**

ATB \[ \rho = 985 - 0.617 \ T(\degree C) \] (A.4)

PFX \[ \rho = 864 - 0.588 \ T(\degree C) \] (A.5)

Bach Ho oil \[ \rho = 830 - 0.62 \ T(\degree C) \] (A.6)

Gippsland \[ \rho = 810 - 0.71 \ T(\degree C) \] (A.7)

*ATB density provided by Syncrude Canada Ltd.

** PFX density provided by Petro Canada Ltd.

*** Bach Ho oil and Gippsland oil density by Saleh (2005)
Appendix B Sample calculation in fouling experiment

Sample calculation for Fouling Experiment, run #17

<table>
<thead>
<tr>
<th>Fluid</th>
<th>% wt ATB</th>
<th>% wt PFX</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>7.56</td>
<td>92.44</td>
</tr>
</tbody>
</table>

Reynolds number calculation

\[
Re = \frac{vd_e}{v} \quad (B. 1)
\]

Where \( v \) is the bulk velocity given by the software based on flow measurement, 0.65 \( (m/s) \)

\( d_e \) is the equivalent diameter, \((d_o - d_i) = (0.0254 - 0.0095) = 0.0159 \ (m)\)

\( v \) is the kinematic viscosity of the mixture, \( (m^2/s) \)

Refutas equation (ASTM Book of Standards, vol.05.04)

\[
VBN_i = 14.534 \ln(ln(v_i + 0.8)) + 10.975 \quad (B. 2)
\]

\[
VBN_{mixture} = \sum_{i=0}^{N} x_i \times VBN_i
\]

*VBN is the viscosity blending number

\[
\nu_{mixture} = e^{\frac{VBN_{mixture} - 10.975}{14.534}} - 0.8 \quad (B. 3)
\]

For ATB at \( T_b = 80 \ ^\circ C \)

\[
\nu(cSt) = 10^{10^{(9.0264 - 3.34584 \ LOG(273.15 + 85))}} - 0.8 = 1469.88 \ (cSt) \quad (B. 4)
\]

\[
VBN_{ATB} = 14.534 \ln(ln(1469.88 + 0.8)) + 10.975 = 39.85
\]
For PFX at $T_b = 80 \ (^\circ C)$

\[ \nu(cSt) = 10^{9.1797 - 3.6653 \log(273.15 + 85)} - 0.8 = 4.11(cSt) \quad (B.5) \]

\[ VBN_{ATB} = 14.534 \ln(\ln(4.11 + 0.8)) + 10.975 = 17.74 \]

From equation B.2 and B.3:

\[ VBN_{mixture} = 0.0756 \times 39.85 + 0.9244 \times 17.74 = 19.41 \]

\[ \nu_{mixture} = e^{(19.41 - 10.975) \div 14.534} - 0.8 = 5.17 \text{ (cSt)} \]

Substituting in equation B.1, Reynolds number will be

\[ Re = \frac{0.65 \times 0.0159}{5.17 \times 10^{-6}} = 1998 \]

**Overall Heat transfer coefficient, Fouling resistance, and Fouling rate at t=6.6 h**

Heating probe power in watts is given by the software and recorded automatically. It could also be calculated using voltage and current.

\[ Q = VI = UA(T_c - T_b) \quad (B.6) \]

\[ U_{t=6.6} = \frac{Q_{t=6.6}}{A(T_c - T_b)} \]

\[ A = \pi d_i L = 3.14 \times 0.0095 \times 0.114 = 0.003402 \ m^2 \]

\[ T_c = 314.2 \ (^\circ C), T_b = 84.85 \ (^\circ C) \]

\[ U_{t=6.6} = \frac{578}{0.003402 \times (314.2 - 84.85)} = 740.71(W/m^2K) \]

To calculate fouling resistance Heat transfer coefficient at t=0, clean heat transfer coefficient, is also needed.
\( U_{\text{clean}} = 742.81 \, (W/m^2K) \) at t=0.3 h

\[
R_f = \frac{1}{U_t} - \frac{1}{U_{\text{clean}}} = \left( \frac{1}{740.71} - \frac{1}{742.81} \right) \times 1000 = 0.003817 \, (m^2K/kW)
\]

Fouling rate calculation is as follows

\[
\frac{dR_f}{dt} = \frac{d}{dt} \left( \frac{1}{U} \right) = \frac{0.003817}{(6.6 - 0.3) \times 3600} = 1.68 \times 10^{-7} \, (m^2K/kJ)
\]

**Film heat transfer coefficient calculation**

\[
Q = VI = hA(T_s - T_b)
\]  \hspace{1cm} \text{(B. 7)}

\( T_s \) is the surface temperature which based on Wilson plot method is calculated from equation 4.6, as follows:

\[ T_{\text{surface}} = T_{\text{core}} - 0.15 \, Q \]

\[ = 314.2 - 0.15 \times 578 = 227.5(\degree C) \]

\[
h = \frac{Q}{A(T_s - T_b)} = \frac{578}{0.003402 \times (227.5 - 84.85)} = 1190 \, \left(\frac{W}{m^2K}\right)
\]
Appendix C Inner wall shear stress calculations

Inner wall shear stress calculation for Bach Ho oil

Meter and Bird (1961) equation for the inner wall shear stress inside an annulus and valid for any flow is as follows (equation 4.15):

\[
\tau_{in} = \frac{r_{out} \Delta p}{2L} \left( \frac{a^2 - \lambda^2}{a} \right)
\]

\[a = \frac{r_i}{r_o} = 0.00515/0.0125 = 0.412\]

\[\lambda = \left( \frac{1 - a^2}{2 \times \ln \left( \frac{1}{a} \right)} \right)^{0.5} = \left( \frac{1 - 0.412^2}{2 \times \ln \left( \frac{1}{0.412} \right)} \right)^{0.5} = 0.6842\]

Pressure drop from Taborek’s (1997) equation for annular flow (equation 4.19)

\[\Delta p = \left[ f \left( \frac{\mu_b}{\mu_w} \right)^{-0.14} \left( \frac{L}{D_e} \right) + k \right] \left[ \frac{Dv_k^2}{2} \right]\]

At \( v = 0.4 \text{ (m/s)}, Re = 2508 \) and \( f \) will be the larger between:

\[f = \frac{64}{Re} = 0.0255\]

\[f = (0.79 \ln(Re) - 1.64)^{-2} = 0.0484\]

\(T_b = 80 \text{ (°C)}, T_w = 240 \text{ (°C)}\)

\[\left( \frac{\mu_b}{\mu_w} \right)^{-0.14} = \left( \frac{\ln(1/(80 + 39.43) + 8.4980) - 2.1390)}{\ln(1/(240 + 39.43) + 8.4980) - 2.1390)} \right)^{-0.14} = 0.9492\]

\[k = 0.5 \text{ from Figure 4.19}\]

\[\rho = 830 - 0.62 \text{ T(°C)} = 830 - 0.62 \times 80 = 780 \text{ (kg/m}^3)\]

\[\Delta p = \left[ 0.0484 \times 0.9492 \left( \frac{0.1016}{0.0147} \right) + 0.5 \right] \left[ \frac{780 \times 0.4^2}{2} \right] = 41.66 \text{ (Pa)}\]
\[ \tau_{in} = \frac{r_{out} \Delta p}{2L} \left( \frac{a^2 - \lambda^2}{a} \right) = \frac{0.0125 \times 41.66}{2 \times 0.1016} \left( \frac{0.412^2 - 0.6842^2}{0.412} \right) = 1.8565 \ (Pa) \]
Appendix D Hot filtration results for a mixture of ATB and Paraflex by Alex Young

To examine the relationship between the concentrations of suspended solids in Atmospheric Tower Bottom (ATB) solutions in heptane, toluene, and paraflex to the temperature of the solution, and the time elapsed since the solution was prepared; to compare the solubility parameters of ATB in heptane, toluene, paraflex, and voltesso, hot vacuum filtration experiment on different solutions with different compositions were done.

Figure D. 1 shows that, at 60 C, ATB in toluene had the lowest concentration of precipitants by volume upon filtration. ATB in paraflex had a much greater concentration of precipitants by volume, and ATB in heptane had a slightly higher concentration of precipitants than the paraflex solution. Thus, ATB is most soluble in toluene, second most soluble in paraflex, and least soluble in heptane.

![](image)

**Figure D. 1 Comparison of precipitant concentrations at 60 C**

Figure D. 2 show the effect that the amount of time elapsed between solution preparation and solution filtration had on the concentration of precipitants in heptane and paraflex solutions.
Paraflex was analyzed at both 60 C and 80 C, and Figure D. 2 shows a comparison.

Figure D. 2 Comparison of precipitant concentrations in paraflex

*(Taken from his Report “ATB Solution Hot Filtration Insolubles Research” (Summer 2014))
Appendix E

Run#5

Table E. 1 Run#5 conditions

<table>
<thead>
<tr>
<th></th>
<th>#5</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fluid % wt ATB</td>
<td>7.4</td>
</tr>
<tr>
<td>% wt PFX</td>
<td>92.6</td>
</tr>
<tr>
<td>Duration (h)</td>
<td>40</td>
</tr>
<tr>
<td>Probe Power(W)</td>
<td>197</td>
</tr>
<tr>
<td>Bulk Temperature(°C)</td>
<td>80</td>
</tr>
<tr>
<td>Probe Initial Temperature (°C)</td>
<td>380.1</td>
</tr>
<tr>
<td>Fluid Velocity (m/s)</td>
<td>0.31</td>
</tr>
<tr>
<td>Re</td>
<td>1100-1200</td>
</tr>
<tr>
<td>Probe Final Temperature (°C)</td>
<td>403.5</td>
</tr>
<tr>
<td>Deposit Thickness (mm)</td>
<td>0</td>
</tr>
</tbody>
</table>
Figure E. 1 Run #5 properties vs. time * Probe temperature on the graph is the core temperature
Run#7

Table E. 2 Run#7 conditions

<table>
<thead>
<tr>
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</tr>
</thead>
<tbody>
<tr>
<td>Fluid</td>
<td></td>
</tr>
<tr>
<td>% wt ATB</td>
<td>7.6</td>
</tr>
<tr>
<td>% wt PFX</td>
<td>92.4</td>
</tr>
<tr>
<td>Duration (h)</td>
<td>48</td>
</tr>
<tr>
<td>Probe Power (W)</td>
<td>223</td>
</tr>
<tr>
<td>Bulk Temperature(°C)</td>
<td>95.2</td>
</tr>
<tr>
<td>Probe Initial Temperature (°C)</td>
<td>415.8</td>
</tr>
<tr>
<td>Fluid Velocity (m/s)</td>
<td>0.6</td>
</tr>
<tr>
<td>Re</td>
<td>1950-2050</td>
</tr>
<tr>
<td>Probe Final Temperature (°C)</td>
<td>429</td>
</tr>
<tr>
<td>Deposit Thickness (mm)</td>
<td>0</td>
</tr>
</tbody>
</table>
Figure E. 2 Run #7 properties vs. time * Probe temperature on the graph is the core temperature
Run#9

**Table E. 3 Run#9 conditions**

<table>
<thead>
<tr>
<th>Fluid</th>
<th>% wt ATB</th>
<th>#9</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>15</td>
</tr>
<tr>
<td>% wt PFX</td>
<td></td>
<td>85</td>
</tr>
</tbody>
</table>

| Duration (h)   | 72       |
| Probe Power(W)  | 247      |
| Bulk Temperature(°C) | 95 |
| Probe Initial Temperature (°C) | 455 |
| Fluid Velocity (m/s) | 0.5 |
| Re              | 1600-1700 |
| Probe Final Temperature (°C) | 465 |
| Deposit Thickness (mm) | 0 |
Figure E. 3 Run #9 properties vs. time * Probe temperature on the graph is the core temperature
Run #11

Table E. 4 Run#11 conditions

<table>
<thead>
<tr>
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<th>#11</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fluid % wt ATB</td>
<td>15</td>
</tr>
<tr>
<td>% wt PFX</td>
<td>85</td>
</tr>
<tr>
<td>Duration (h)</td>
<td>48</td>
</tr>
<tr>
<td>Probe Power (W)</td>
<td>370</td>
</tr>
<tr>
<td>Bulk Temperature (°C)</td>
<td>90</td>
</tr>
<tr>
<td>Probe Initial Temperature (°C)</td>
<td>570</td>
</tr>
<tr>
<td>Fluid Velocity (m/s)</td>
<td>0.5</td>
</tr>
<tr>
<td>Re</td>
<td>1690-1800</td>
</tr>
<tr>
<td>Probe Final Temperature (°C)</td>
<td>607</td>
</tr>
<tr>
<td>Deposit Thickness (mm)</td>
<td>0</td>
</tr>
</tbody>
</table>
Figure E. 4 Run #11 properties vs. time * Probe temperature on the graph is the core temperature
## Run#13

**Table E. 5 Run#13 Conditions**

<table>
<thead>
<tr>
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<th>#13</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fluid % wt ATB</td>
<td>15</td>
</tr>
<tr>
<td>Fluid % wt PFX</td>
<td>85</td>
</tr>
<tr>
<td>Duration (h)</td>
<td>26</td>
</tr>
<tr>
<td>Probe Power(W)</td>
<td>412</td>
</tr>
<tr>
<td>Bulk Temperature(°C)</td>
<td>90</td>
</tr>
<tr>
<td>Probe Initial Temperature (°C)</td>
<td>640</td>
</tr>
<tr>
<td>Fluid Velocity (m/s)</td>
<td>0.5</td>
</tr>
<tr>
<td>Re</td>
<td>1800-1900</td>
</tr>
<tr>
<td>Probe Final Temperature (°C)</td>
<td>650</td>
</tr>
<tr>
<td>Deposit Thickness (mm)</td>
<td>0</td>
</tr>
</tbody>
</table>
Figure E. 5 Run #13 properties vs. time * Probe temperature on the graph is the core temperature