

DEPOSITION FROM CRUDE OILS IN HEAT EXCHANGERS

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ABSTRACT

Deposition in flow lines, processing and heat transfer equipment arises from fouling species which may either be present in the fluid or be generated in the vicinity of the equipment surface. Recent research on deposition during heat transfer from petroleum feedstocks is reviewed. For low-sulphur light crude oils, deposition is largely due to particulates and gums. For medium-sulphur crude oils, formation of iron sulphides plays a major role in deposition. In unstable heavy oil systems, suspended asphaltene are the fouling species. Trace quantities of impurities such as dissolved oxygen or suspended corrosion products add markedly to deposit formation. The influences of flow velocity, bulk and surface temperatures and particulate concentrations are demonstrated through experimental results, and compared to expectations from simple models. Through understanding of the key steps in the deposition processes a rational mitigation strategy can be formulated.

INTRODUCTION

The build-up of undesirable material on the surface of process equipment such as heat exchangers generally involves the deposition of solid or semi-solid foulant species which are either already present in the fluid or are created from precursors by physical or chemical processes as the fluid passes through the equipment. Although precursor concentrations may be very low, large amounts of deposit may form where flow-rates are high. For example, in a 300,000 bbl/d oil refinery, if one part per million by weight of the oil is deposited in the pre-heat exchanger train, in one year about 15 tonnes of solid carbonaceous deposits will have accumulated. This insulating layer, which preferentially forms on the heat transfer surface, will reduce the rate of heat

transfer, lowering the temperature at the exit of the preheat train exchangers. Since the temperature of oil entering the downstream distillation column must be maintained, any loss due to fouling in the preheat train exchangers must be made up in the furnace, usually by increasing the flowrate of furnace fuel. The deposits on the heat exchanger walls therefore represent several challenges to plant engineers:

1. The decrease in heat transfer efficiency ultimately results in consumption of additional fuel gas leading to increases in greenhouse gas emissions.
2. Pressure drop through the equipment increases, raising pumping power needs.
3. Exchangers may have to be taken out of service for cleaning, leading to production losses.
4. Disposing of the carbonaceous deposits which can contain sulphur, nitrogen and metals creates additional environmental problems.

Hence minimizing deposit formation is a key step in operating processes in an efficient and environmentally sensitive manner. This is particularly true in the above case of crude oil preheat train fouling, since the crude oil distillation unit is the largest consumer of thermal energy among distillation processes in chemical and petroleum industries (Panchal and HuangFu, 1998).

In order to use rational means to minimize fouling, it is essential to identify the precursor species, determine how and where the foulant is formed, and what governs its accumulation on the surface. Figure 1 shows a multi-step chemical fouling mechanism (Watkinson and Wilson, 1997). Here, soluble Precursor A which enters with the incoming fluid may transport to the surface and undergo surface reaction to produce insoluble Deposit C on the wall. Alternatively, Precursors A may form insoluble Foulant B by

reaction in the bulk fluid or in the thermal boundary layer and then Foulant B transport and adhere to the surface. Foulant B may undergo an ageing reaction on the surface to produce Deposit C. For example, the precursors could be a reactive olefin and traces of oxygen which could undergo bulk reaction to form an insoluble polymeric peroxide gum foulant, which could transfer and adhere to the heat transfer surface.

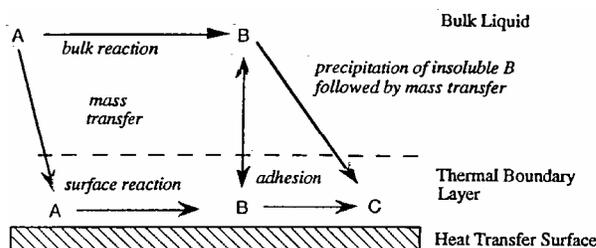


Fig. 1 Multi-step Fouling Mechanism (Watkinson and Wilson, 1997)

For many cases of fouling, the precursor may be known. For example, suspended silt particulates may be the foulant. In processing complex organic mixtures such as crude oils, or heavy oils and their downstream products, the identification of precursors and foulants is itself a challenge. For crude oil streams, a number of root causes of fouling may be operative:

- *suspended impurities such as dirt, clay or rust attach to surface
- *insoluble gum forms in the bulk oil due to oxygen ingress from storage tanks
- *asphaltenes or resins precipitate due to changes in composition, temperature, or pressure
- *soluble sulphur species react with tube surface
- *oil components undergo thermal decomposition forming coke.

In addition to understanding foulant formation rates in different environments, the effects of process variables such as bulk and surface temperature, flow velocity, and composition on deposition rates must be determined. In this paper results of some recent studies on oil fouling are presented to identify the cause of fouling and to provide guidance for mitigation.

APPARATUS, PROCEDURES AND CALCULATIONS

Fluid is re-circulated through an electrically heated annular probe, operated at constant heat flux (Watkinson et al., 2000).

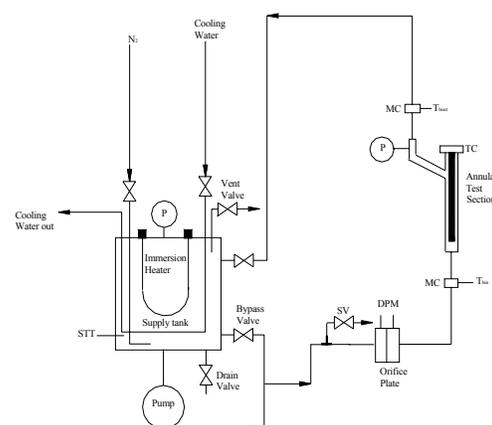


Fig. 2 Re-circulation fouling loop; MC-mixing chamber, DPM-differential pressure monitor, STT-supply tank temperature, SV-sampling valve

Flowrate, pressures, temperatures of probe wall and bulk fluids, probe current and voltage are monitored with time.

The thermal fouling resistance reflects the decline in heat transfer coefficient with time, and is calculated from the increase in surface temperature:

$$R_f = 1/U - 1/U_o = \{(T_s - T_b) - (T_s - T_{b,o})\} / q \quad (1)$$

After an experiment, the carbonaceous deposit is recovered from heated section of probe. The rate of fouling, or rate of increase of thermal resistance ($1/U$), is related to deposit thickness, and deposit mass per unit area, as given below:

$$[dR_f/dt] = d[1/U]/dt = [dx/dt]/k_f = [dm/dt]/\rho_f k_f \quad (2)$$

In a laboratory system where fluid is re-circulated, foulant can be created over time by reactions in the bulk or wall-regions, changing the fluid composition. For such cases, the initial fouling rate $[dR_f/dt]_{t=0}$ before the composition has changed, is of key interest. To minimize the role of bulk fluid reactions in changing precursor or foulant concentrations, bulk temperatures may be maintained significantly below surface temperatures.

Oils cover a range of properties, as outlined in Table 1. Light sweet crude oils, medium sour crude oils and blends of heavy oils with aliphatic or aromatic diluents have been studied.

RESULTS AND DISCUSSION

Suspended Impurity Effects in Oil Fouling

Crude Oil SSB initially contained about 15-40 ppmw (parts per million by weight) of insoluble solids as measured by filtration through a 1-micron pore size filter. Fine inorganic particulates were added to the oil sample which was subsequently decanted after one hour of settling time. This procedure resulted in suspended particle additions to 250 ppmw in the oil. The fouling resistance versus time was measured (Watkinson et al., 2000), with results shown in Figure 3. Both iron and aluminum oxides added dramatically to the growth of the fouling resistance, yielding an order of magnitude increase in fouling rate with a similar increase in suspended solids content.

Table 1 Properties of Crude Oils and Heavy Oil Blends

Type	Oil	% As	% Insol	% S	ρ g/ mL	μ 20°C mPa.s
Light	SSB	0.2	0.002	0.09	0.87	5
	GPS	0.35	0.24	0.12	0.79	2
	CSK	0.08	0.15	0.12	0.79	1.8
Med.	LSB	3.1	0.13	1.3	0.85	13
	BHO	1.5	0.47	0.03	0.82	22
	FO	0.6	0.006	0.66	0.85	24
Heavy Blend	ATB/ /PFX	1.4	0.54	0.45	0.88	28
	ATB/ HVGO	1.4	0.10	1.33	0.89	16
	VR/ PFX	1.8	1.1	0.49	0.89	16
	VR/ HVGO	1.8	0.16	1.37	0.89	15
	HO/ FO	1.6	0.3	0.48	0.87	30

As= asphaltenes; Insol. = hot filtration insolubles; SSB = Syncrude Sweet Blend; CSK = Cossack; GPS=Gippsland; LSB=Light Sour Blend; BHO=Bach Ho; ATB= Athabasca Atm. Topped bitumen; VR= Cold Lake Vac. Residue; HO= Cold Lake Heavy Oil; FO= Fuel Oil; PFX=Paraflex; HVGO=Heavy Vac. Gas oil.

In a fouling run where fluid is re-circulated and no generation of foulant particles occurs, the decrease in suspended particle concentration over the duration of the run should reflect the extent of fouling. Saleh et al. (2005) have shown that indeed for runs of the same duration, low fouling rates correspond to small decreases in suspended solids concentrations, and high fouling rates correspond

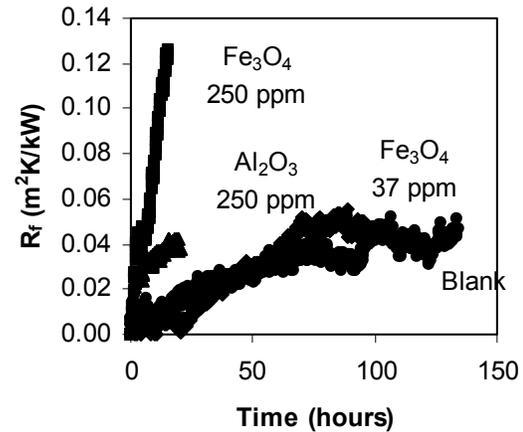


Fig. 3 Fouling Resistance versus Time with SSB Oil and Particulate Additions ($T_b = 155^\circ\text{C}$, $T_{s,o} = 290^\circ\text{C}$, $V = 0.44\text{m/s}$)

to large decreases. A sample of BHO crude oil which initially contained about 0.5 wt % gum-like solids, was filtered through a 1-micron filter, and fouling rate of the fluid compared with and without filtration. Results shown in Figure 4 (Saleh, 2006)

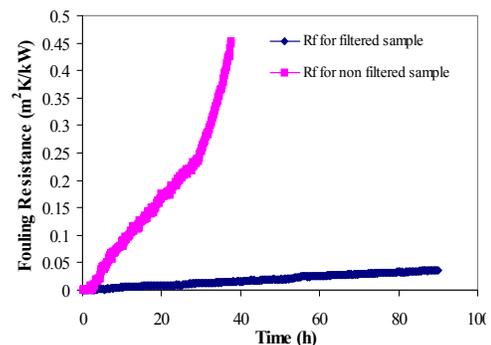


Fig.4 Effect of Removal of Suspended Solids by Pre-filtration on Fouling of BHO Oil at $T_b = 80^\circ\text{C}$, $T_{s,o} = 240^\circ\text{C}$, $V = 0.25\text{m/s}$, $P = 379\text{kPa}$.

indicate that near elimination of suspended solids can reduce extent of fouling to close to detection levels. Hence Figures 3 and 4 illustrate the key role of suspended particles in oil fouling for cases where the particles are present as suspended impurities in the feed oil.

Insoluble Gum Formation in Organic Fluid Fouling

In refineries, small amounts of oxygen can enter oil streams via leaks into storage tanks or in vacuum processes. Auto-oxidation processes can then lead to formation of soluble and insoluble gums which deposit on heat exchange equipment. A simplified reaction scheme, which illustrates the process but does not represent the detailed mechanism is credited to W.F. Taylor, in Braun and Hausler (1977):

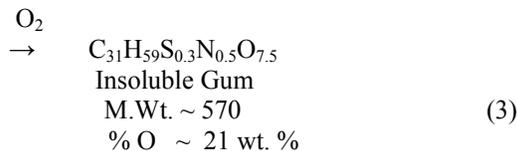
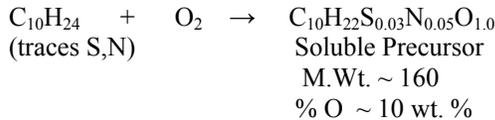


Figure 5 shows that saturating SSB Crude Oil with air as opposed to nitrogen at 155°C, raises its rate of fouling by an order of magnitude.

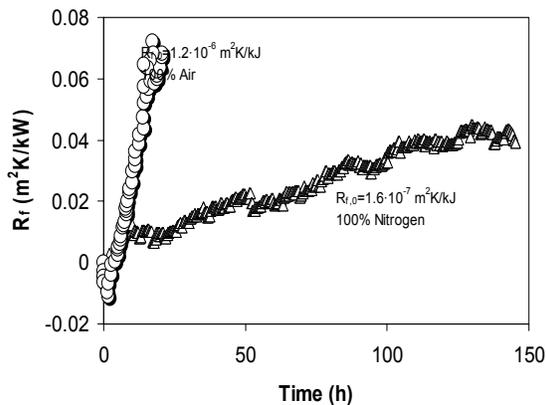


Fig.5 Fouling resistance versus time for SSB Oil with Nitrogen and Air Saturation at $T_b = 155^\circ\text{C}$, $P=377\text{kPa}$ (Watkinson et al., 2000)

Figure 6 indicates that even low concentrations of dissolved oxygen below 10 ppmw in the oil (determined using a GC-MS method) can have significant effects on crude oil fouling through gum formation. Co-incident with the presence of significant levels of dissolved oxygen is an increase of suspended gum particle concentrations measured by hot filtration (Watkinson et al., 2000).

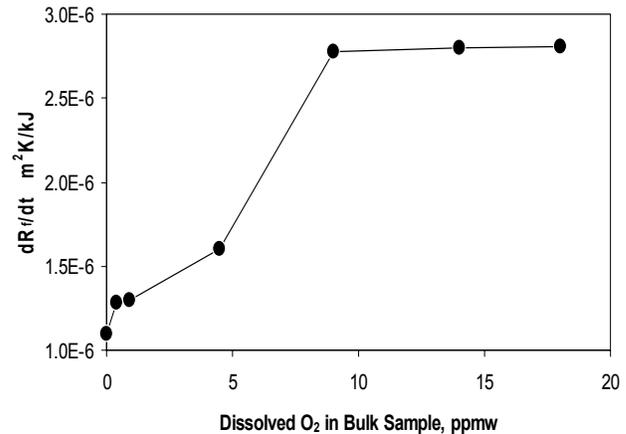


Fig.6 Effect of Dissolved Oxygen on Initial Fouling rate of SSB Crude Oil at $T_b = 75^\circ\text{C}$, $T_{s,o} = 290^\circ\text{C}$, $V = 0.44\text{ m/s}$ (Watkinson et al., 2000)

Both gum particles and deposits indicate high levels of oxygen content typical of insoluble gums (Table 2), with (O/C) atomic ratios about 0.1. Hence the dissolved oxygen generates insoluble gum particles which end up in the heat exchanger deposits. This type of reaction and its effect on fouling has been studied in depth using model compounds such as indene and di-cyclopentadiene which react with oxygen to produce polymeric peroxide species (Wilson et al., 1995).

Asphaltene Deposition in Oil Fouling

Solvent fractionation of petroleum provides a useful framework to understand deposition from crude oils (Mushrush and Speight, 1995). Crude oil can be considered as a colloidal system made up of: Asphaltenes-the heaviest and most polar fraction (Mol. Mass ~ 2000); Resins- a dispersant for the asphaltenes (Mol. Mass ~ 1500); Small-ring Aromatics-a solvent for the asphaltenes; and the Saturates-a precipitant or non-solvent for the asphaltenes. Precipitation of asphaltenes, which can be defined as the fraction soluble in toluene and insoluble in heptane, is a major cause of fouling. When composition, temperature or pressure changes de-stabilize the colloidal system, some asphaltenes precipitate from the oil as a second phase. The Colloidal Instability Index (Asomaning and Watkinson, 1999), C.I.I.,

$$\text{C.I.I.} = \frac{[\text{Asphaltenes} + \text{Saturates}]}{[\text{Resins} + \text{Aromatics}]} \quad (4)$$

represents a measure of instability. When C.I.I. < 1.0, the amount of resins plus aromatics is sufficient to maintain the asphaltenes in “solution”. Addition of saturates or removal of aromatics can shift the oil composition such that C.I.I. > 1.0, and asphaltenes will precipitate.

Two heavy oil cuts were used in recent studies (Hong E, 2005): Cold Lake Vacuum Residue (18 % asphaltenes, C.I.I. = 0.4), and Athabasca Atmospheric Topped Bitumen (13 % asphaltenes C.I.I. = 0.53). Each stable oil cut was blended with Paraflex (99 % Saturates), and with a Heavy Vacuum Gas Oil (68 % Saturates), and various mixtures. Figure 7 shows that with 10 % wt. Cold Lake Vacuum Residue in a binary blend with Paraflex, extensive precipitation and fouling occurs, whereas with HVGO as the diluent, precipitation is minimal and extent of fouling is very low. Similar results were obtained with the Athabasca ATB.

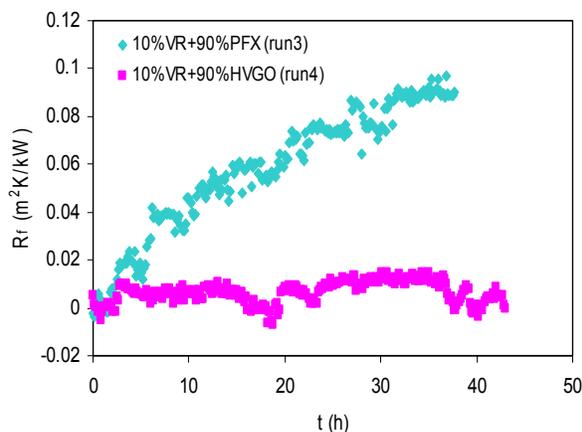


Fig.7 Asphaltene Fouling of Cold Lake Vacuum Residue Blended with Paraflex versus Heavy Vac. Gas Oil (VR-PF: $C_{sas} = 9.1$ g/L, CII=12;VR-HVGO: $C_{sas} = 1.4$ g/L, CII = 1.9)E. Hong (2005)

Figure 8 shows that for fixed velocity and temperatures in the bulk and on the clean surface, fouling rates of VR with various HVGO/PFX blends at a given asphaltene concentration can be simply correlated with their C.I.I. values. Models based on solubility parameters provide a less empirical basis for instability (Wiehe and Kennedy, 2000) and have been found to be applicable to thermal fouling (Al-Atar and Watkinson, 2002). For these unstable oils, the precipitated asphaltene solids have compositions similar to those of the deposits (Table 2), and the fouling rate increases strongly with suspended asphaltene concentrations.

Thus asphaltene fouling is another case of particulate deposition, but is complicated by precipitation and agglomeration processes.

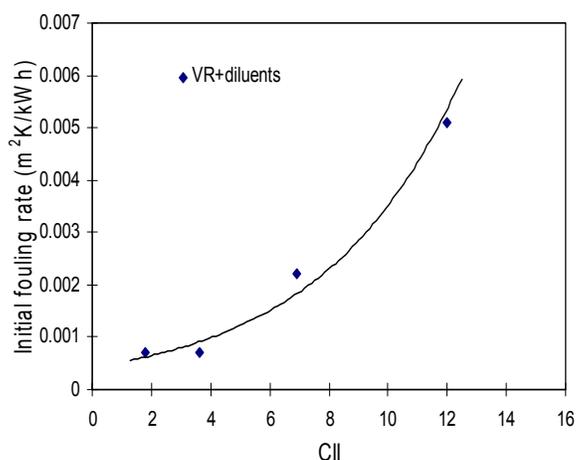


Fig. 8 Fouling Rate of Heavy Oil Blends versus C.I.I at Asphaltene Content of 1.77 wt.% $T_b=85^\circ\text{C}$, $T_{s,o} = 260^\circ\text{C}$, $V=0.75$ m/s. (E. Hong 2005).

Sulphur-Containing Crude Oils

Experiments were run with sour crude oils (Srinivasan and Watkinson, 2005). During the course of 48-hour experiments at high temperatures ($T_b \sim 270^\circ\text{C}$, $T_{s,o} \sim 370^\circ\text{C}$) the oils increased in dissolved metals content due to corrosion in the heat exchanger loop. For LSB oil (Table 1), which contained 1.3 %S, deposits were rich in iron sulphide content (Table 2). At the highest temperatures, deposits were about 30 % organic, and the remainder inorganic. The mechanism of such corrosion-related fouling has been discussed by Braun and Hausler, (1977), however much remains to be learned about the interaction of corrosion and fouling.

Table 2. Composition of Deposits and Insoluble Solids

Oil	% Ash	%C	%H	%S	H/C	O/C
SSB-P	6.6	68.4	5.9	2.0	1.0.	0.11
SSB-D	7.8	76.3	6.9	2.8	1.0	0.09
GPS-P	0.75	92.4	5.5		0.71	
GPS-D	2.5	76.5	7.8	2.9	1.22	
LSB-D	78	10	1	20	1.2	%Fe=48
BHO-P	1.9	86.8	7.2		1.0	
BHO-D	4.9	80.5	7.6	2.6	1.13	
VR/PF-P	4.3	80.6	7.4	6.1	1.1	
VR/PF-D	2.2	81.9	7.2	5.2	1.06	

D = deposit , P = filtered particulates

Transport Plus Adhesion Models

Table 3 summarizes the initial rate equations for a number of available fouling models. In some cases the equations have been simplified to provide for direct comparison.

For the complex case of crude oil fouling, the model parameters are not predictable, nevertheless, the effect of process parameters on fouling rate can be rationalized.

Table 3 Simplified Models for Initial Rate of Crude Oil Fouling

Watkinson, Epstein (1970)	$dR_f/dt = a \exp(-E/RT_s) / V^n$ (5)
Epstein, (1 st Order, Simplified) (1997)	$dR_f/dt = C_b / [a/V + bV^2 \exp(-E/RT_s)]$ (6)
Ebert, Panchal (1997)	$dR_f/dt = a \exp(-E/RT_f) / Re^n - bV^2$ (7)
Yeap et al. (2004)	$dR_f/dt = 1/[a/V T_s^{2/3} + bV^2 \exp(-E/RT_s)] - cV^{0.8}$ (8)

For example, fouling due to particles can be modelled as two steps in series, in which suspended species B is transported to the wall region, followed by an adhesion step in which B becomes attached to the surface (Epstein, 1997). Where transport of species to the surface dictates the overall rate of fouling, fouling rates should increase weakly with surface and bulk temperature, and increase with velocity nearly linearly in turbulent flow and more weakly in laminar flow. When adhesion of the foulant to surface layer controls the rate, fouling should increase strongly

with surface temperature because of the temperature sensitivity of the adhesion process, more weakly with bulk temperature, and decrease with increases in velocity due to the reduced contact time for adhesion to occur. These two steps are reflected in the two terms of the denominator of equation (6), Table 3. Although much of the present data was taken in the transition region rather than in turbulent flow, the general trends of velocity and temperature predictions for the two steps can be tested.

Figures 9 and 10 show that for all six oils tested, the initial fouling rate decreases with increasing velocity (at a fixed surface temperature). This includes light and medium oils where fouling is caused by particulates, including solids initially present in the oil (GPS, CSK, BHO) and extra solids generated by autoxidation (SSB), as well heavy oil blends with suspended asphaltenes (HOP). A similar trend is also found for the medium sour oil (LSB) where corrosion played a role. Although the velocity range is limited, and the Reynolds numbers often include some values in the transition region, the decline in rate with increasing velocity suggests that the transport step does not control fouling rates in these cases.

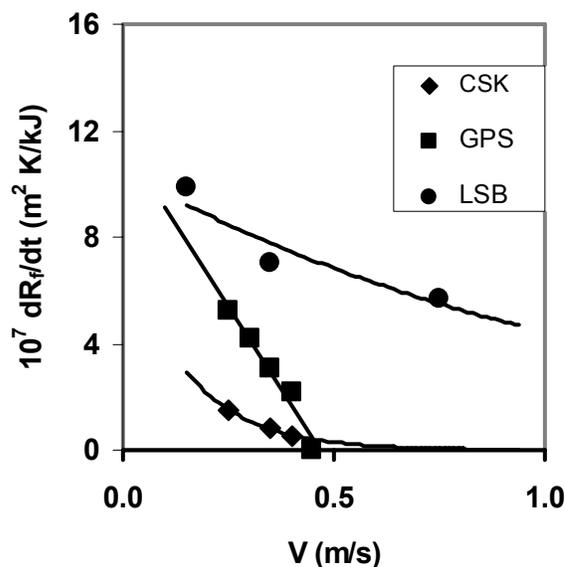


Fig.9 Velocity Effects on Fouling Rates of Cossak, Gippisland and Light Sour Blend

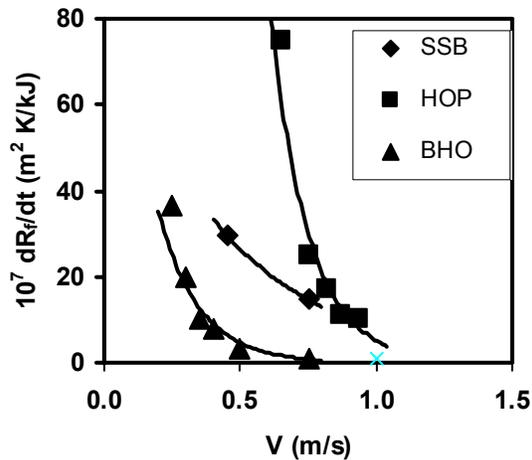


Fig. 10 Velocity Effects on Fouling Rate of Sweet Blend, Bach Ho and Heavy oil-Paraflex blend

Temperature effects on fouling rates are commonly plotted in the “fouling Arrhenius” form where the logarithm of the initial fouling rate is plotted versus the inverse of the absolute surface or film temperature (Figures 11,12). The film temperature, determined from averaging bulk and surface temperature, reflects the temperature in the thermal boundary region near the surface where reaction and adhesion processes may occur. Although widely used, it is not necessarily the best parameter to correlate fouling rates in all situations. For asphaltenes, the solubility increases with temperature. Hence the suspended asphaltene concentration, decreases with increasing bulk temperature. This leads to a decrease in fouling rate with bulk fluid temperature at a given surface temperature. Thus, raising the film temperature by increasing bulk temperature will decrease fouling rates under this condition. By contrast, for particulate fouling due to other suspended impurities such as corrosion products or gum particles, the solubility of the foulant does not change markedly with bulk temperature, and fouling rates will increase with bulk temperature at a given surface temperature. For this latter situation the film temperature provides a good average value with which to correlate fouling rates.

In all cases shown in Figures 11 and 12, temperature dependence is significant, leading to fouling activation energies ranging from 35-95 kJ/mol. Fouling activation energies require care in

interpretation, since they reflect the overall multi-step fouling process, and are influenced in magnitude by whether they are based on film or surface temperature, and possibly by flow effects. Nevertheless, this magnitude of temperature dependence is high for a transport process, and hence suggests that a reaction process or adhesion on the surface governs the fouling rates for these cases. Table 4 summarizes data for the present oils, and a few other cases from recent work.

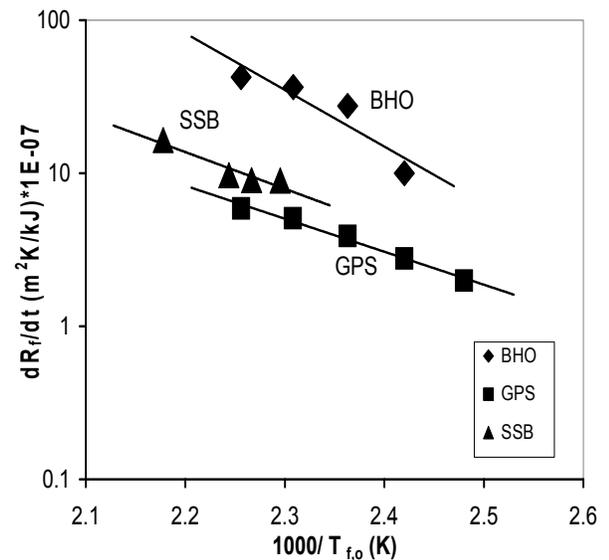


Fig.11 Initial fouling rate as a function of film temperature for three oils

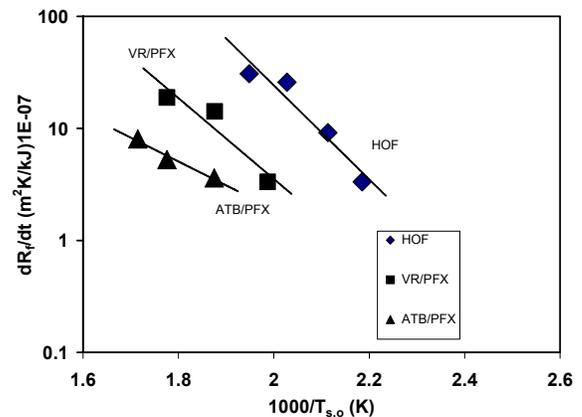


Fig. 12 Initial fouling rate as a function of surface temperature for asphaltene fouling.

The weakest temperature dependence of the fouling rate ($E_f < 42$ kJ/mol) occurred for the lighter crudes under nitrogen, where the rates were lowest (2-10 E-07 m²K/kJ), and the foulant appeared to be suspended particulates. For medium oils, rates were somewhat higher (9-40 E-07 m²K /kJ), as were fouling activation energies (60-84 kJ/mol). For blends which contained heavy

Table 4 Summary of Fouling Results at $T_b \sim 85^\circ$ C.

Oil* Type	Oil	Max $T_{s,0}$ (°C)	Max dR_f/dt 10^{-7} m ² K/kJ	** E_f kJ/mol	Max Re	Foul ant [†]
L	SSB	270	10	20	4100	P
L	CSK	260	2	35	3800	P
L	GPS	260	6	42	4500	P
L	SSB-O2	297	28	56	4800	G
M	LSB	370	10	59	5600	FeS/As
M	BHO	260	42	72	3000	As
M	FO	320	9	84	4700	As
HB	ATB, VR/PFX	290	20	95	2400	As/PFX
HB	5% HO/FO	245	34	81	3000	As/FO
HB	10% HO/FO	220	32	184	1850	As/FO
HB	10% HO/FO-O2	220	330	212	1850	As/FO

*L=Light, M=Medium, HB=Heavy Blend

**based on film temperature

↓ P=particles, G=gum, As= asphaltenes

oils, fouling was due to asphaltene or asphaltene plus oils, and both the fouling rates (20-34 E-07 m²K/kJ) and fouling activation energies were high (80-184 kJ/mol). With both the light oil SSB, and the 10% HO-FO blend, oxygen ingress dramatically raised both the fouling rates, and the temperature dependence of the fouling process for the oil in question. Based on mechanisms indicated in Figure 1, it appears that whenever particulates already present in the oil stream cause fouling, the temperature dependence will be relatively low, reflecting an adhesion process, such as with the light crude oils in Table 4. Where temperature dependent reactions or precipitation processes generate insolubles or deposits, temperature dependence is higher, and rates can become much greater, as for the medium and heavy

blends of Table 4. In the most extreme case tested, where heavy oil/fuel oil blend is saturated with air at several atmospheres (Asomaning and Watkinson, 2000), fouling rates reach hundreds of times that for light crude oils, and activation energies are almost an order of magnitude higher.

CONCLUDING REMARKS

Fouling in crude oil systems can have a significant effect on fuel consumption in a refinery, and hence on green-house gas emissions. Deposition from multi-component organic mixtures such as crude oils can arise from several causes. Fouling rates are complex functions of oil composition, temperature, velocity and particulate content

For lighter crude oils at relatively low temperatures, fouling is related to concentration of particulate matter (silt, corrosion products, etc.), shows a relatively weaker temperature dependence and can be reduced by high velocities and removal of particulates. Gum formation due to oxygen ingress, and asphaltene precipitation during dilution or blending, lead to insoluble organic phases which give rise to fouling rates that are relatively high and more strongly surface-temperature dependent. Rates may be controlled by rigorous exclusion of oxygen and maintaining low C.I.I. levels in blending respectively. Sulphurous crudes give rise to deposits which are high in iron sulphide content.

A range of velocity effects and temperature sensitivities on the fouling rate has been observed. Although care is required in interpretation of these results, trends of effects of velocity, and temperature suggest that adhesion of the foulant to the surface rather than transport of foulant governs the fouling process for the oils tested.

Acknowledgement

Ongoing support from the Natural Sciences and Engineering Research Council of Canada, and specific project support from several industrial sources is acknowledged.

Nomenclature

a,b,c	constants in model equations
C	concentration of precursor (g/m ³)
D _e	equivalent diameter for annulus (m)
E	fouling activation energy (kJ/mol)
k _f	deposit thermal conductivity (W/mK)
m	deposit mass/area (g/m ²)
n	velocity exponent

Q	heat flow (W)
q	heat flux (W/m ²)
R	gas constant (J/mol K)
R _f	thermal resistance due to fouling (m ² K/kW)
Re	Reynolds number based on D _c and T _b
t	time (s)
T _b	fluid bulk temperature (°C, or K)
T _f	film temperature = 0.5[T _b + T _s] (°C, K)
T _s	surface temperature (°C, K)
U	heat transfer coefficient (kW/m ² K)
V	bulk velocity (m/s)
x	deposit thickness (m)

Subscripts

b	bulk
f	film, foulant
s	surface
sas	suspended asphaltenes
o	at time zero (clean condition)

Greek

ρ	density (g/m ³)
μ	viscosity (mPa.s)

Acronyms

ATB	Athabasca atmospheric topped bitumen
BHO	Bach Ho oil
CII	Colloidal Instability Index
CSK	Cossack oil
FO	Fuel oil cut
GPS	Gippsland oil
HO	Cold Lake Heavy Oil
HOF	Heavy oil/fuel oil blend
HVGO	Heavy vacuum gas oil
LSB	Light sour blend oil
PFX	Paraflex solvent
SSB	Syncrude sweet blend oil
VR	Cold Lake Vacuum Residue

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