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PILOT PLANT THERMAL HYDROCRACKING OF GCOS (GREAT CANADIAN OIL SANDS) BITUMEN: 1. THE EFFECT OF HEAVY OIL RECYCLE ON THE PLANT OPERATION

C.P. Khulbe, B.B. Pruden, J.M. Denis and W.H. Merrill
Canadian Fossil Fuel Research Laboratory,
Petroleum Processing Section

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by

C.P. Khulbe*, B.B. Pruden*, J.M. Denis** and W.H. Merrill***

ABSTRACT

The objective of the present investigation was to determine the operability of the thermal hydrocracking plant using a recycle-oil stream to increase liquid velocity in the reactor, in an attempt to reduce sludge or coke formation. Life runs of about three weeks were conducted with heavy-oil recycle fed to the bottom of the reactor. The two long experimental runs in this series, R-2-1-2 and R-2-2-4, are described in detail. All shorter runs in which recycling of heavy oil was attempted are also described. Of main concern in this report are those factors which influenced the steady operation of the pilot plant, such as coke formation and deposition of metal sulphide which caused fouling and line plugging.

The heavy-oil recycling, at a recycle to feed weight ratio of 2:1 or greater, reduced coke formation in the reactor compared with once-through operations at the same temperature, pressure and pitch conversion. Total recycle of the heavy ends before reaching the required temperature gave a smooth start-up. Adjusting the recycle-oil withdrawal rate was better than adjusting the feed flow as a means of maintaining the hot-catch-pot level. The increase in the ash content of the reactor fluid, coupled with a short "first-pass" residence time are perhaps the reasons for the suppression of coke formation when heavy oil is recycled.

*Research Scientists, ** Head, Process Engineering Section, and ***Manager, Canadian Fossil Fuel Research Laboratory, Energy Research Laboratories, Canada Centre for Mineral and Energy Technology, Department of Energy, Mines and Resources, Ottawa, Canada.

UNE INSTALLATION PILOTE D'HYDROCRAQUAGE
THERMIQUE DE BITUME DE LA GCOS (GREAT CANADIAN OIL SANDS):
1. L'EFFET DU RECYCLAGE D'HUILE LOURDE SUR LE FONCTIONNEMENT DE L'INSTALLATION

par

C.P. Khulbe*, B.B. Pruden*, J.M. Denis** et W.H. Merrill***

SOMMAIRE

L'objectif de cette présente étude visait à connaître la possibilité de recycler de l'huile lourde sur une installation d'hydrocraquage thermique, dans le but de réduire la formation de coke ou de boue par l'augmentation du débit de liquide dans le réacteur. Des opérations d'une durée de trois semaines ont été entreprises avec une huile lourde alimentant le réacteur par le bas. Les deux marches d'essai de cette série, R-2-1-2 et R-2-2-4 y sont décrites plus en détails. On donne, également, la description de toutes autres opérations de courte durée pendant lesquelles des essais de recyclage d'huile lourde ont été effectués. Les principaux facteurs qui nous intéressent ici, sont ceux qui affectent la stabilité du fonctionnement de l'installation pilote, tels la formation du coke et le dépôt de sulfure métallique qui causent l'encrassement et l'obturation des conduits.

Le recyclage d'huile lourde a réduit la formation de coke dans un rapport recyclage/charge d'alimentation de 2:1 en poids dans le réacteur en comparaison avec des essais d'une seule opération à la même température, pression et conversion du brai. Le démarrage fut régulier grâce à un recyclage complet des fractions lourdes avant d'avoir atteint la température désirée. Il a été plus facile de maintenir le niveau du séparateur à haute température en contrôlant le taux de récupération de l'huile recyclée que le débit d'alimentation. Il semblerait que l'augmentation du contenu en cendre du liquide dans le réacteur, de pair avec un court temps de séjour du liquide dans le réacteur pendant la première opération sont responsables de l'élimination de la formation du coke lors du recyclage d'huile lourde.

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* Chercheurs scientifiques, ** Chef, Section de la technique de combustion, et *** Gestionnaire, Laboratoire de recherche sur les combustibles fossiles du Canada, Laboratoires de recherche sur l'énergie, Centre canadien de la technologie des minéraux et de l'énergie, Ministère de l'Energie, des Mines et des Ressources, Ottawa, Canada.

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INTRODUCTION

One of the objectives of CANMET (the Canada Centre for Mineral and Energy Technology) is to develop an economical process for upgrading bitumen and heavy oils. This is in keeping with the Energy Research Program of the Department of Energy, Mines and Resources and its policy of ensuring the effective use of Canada's mineral and energy resources. The thermal hydrocracking process has been developed at the Energy Research Laboratories as a method of upgrading Athabasca bitumen.

This bitumen contains 51.5% by weight of pitch (material boiling above 524^{o}C) and about 0.6% by weight of ash, as shown in Table 1. In converting this pitch to distillable material, there is a tendency to form coke deposits, leading to fouling of equipment such as the reactor and the down-stream hot vapour-liquid separator. This necessarily affects operation of the system and generally leads to lower efficiency and eventual shut-down because of plugging. It is therefore highly desirable to provide a means for inhibiting or materially reducing coke formation in the reactor and thus increasing operability and on-stream time.

In a U.S. patent, Chervenak et al. (1) claimed that in the upflow liquid-phase ebullated bed process the coke-formation tendency of the reactor liquid was related to the hydrogen content of the pitch. If the hydrogen content of this material fell below 6.8 wt %, coke was deposited in the reactor. For a bitumen having less than 1 wt % ash, coke formation in the reactor could be avoided by adding an appropriate hydrogenation catalyst at start-up to establish a substantial consumption of hydrogen. In another U.S. patent, Wolk et al. (2) stated that increasing the ash content in the feed decreases coke deposition. They suggested that high-ash feeds containing about 3.5 wt % are also less likely to lay down coke in the separator than a low-ash feed. Layng (3) claimed that if a small portion of the heavy oil is recycled, thus increasing ash content in the reactor liquid, and the reactor effluents are quenched, a minimum pitch conversion of 60% can be obtained at space velocities between 0.5 and 2 h^{-1} and temperatures of 370-455^{o}C without forming any coke deposits in the reactor or separator. Wolk (4) claimed that the deposition of coke in the hydrogenation of tar-sand bitumen could be avoided by regulating the concentration of ash in the reactor liquid. He suggested that this could be accomplished by feeding the reactor liquid products to a liquid cyclone separation unit and recycling the underflow stream with increased ash concentration to the reactor. It was observed that in regulating the ash concentration in the reactor from about 4 to 10 wt %, the formation of coke was reduced substantially, resulting in improved operability of the hydro-conversion process. A recycle-oil/feed ratio of 0.05 to 1.0 gave conversions of the +975^{o}F fraction of 70 to 85% at about 850^{o}F reactor temperature and 1500 psi pressure.

The experimental work carried out at the Energy Research Laboratories in refining residual oil and tar has been largely concerned with catalytic hydrogenation (5,6,7). The bitumen contains approximately 0.6% ash (clay, chemically bound nickel, vanadium and iron), 4.7% sulphur (Table 1) and 0.4 to 0.6% nitrogen. These constituents make catalytic hydrocracking extremely difficult. High hydrogen pressures minimize many of the problems but the catalyst does become coated with mineral matter and metals which makes regeneration of the catalyst impractical.

A previous report (8) described the pilot plant investigation of thermal hydrocracking of Athabasca bitumen to produce distillate hydrocarbon fractions. Experiments were made in the liquid phase using a conventional-flow apparatus with a bottom-feed reactor and a hydrogen-scrubbing and recycle system. High pressures of 2500-3500 psi improved the operation by suppressing coke and sludge formation and allowed the reaction to be carried out at conditions of over 80% pitch conversion.

In pilot plant runs without heavy-oil recycle, the liquid feed at a liquid hourly space velocity (LHSV) of 2.0, for example, is 9000 g/h for a 4.5 ℓ reactor. The feed contains typically 51.5% pitch or material boiling above 524^{o}C. Again in a typical run at 13.9 MPa (2000 psi) pressure and 460^{o}C temperature, about 76% of the pitch is converted to lower-boiling distillate and gases, and the liquid weight-yield is about 92%. Because of the flow of hydrogen and light hydrocarbon gases in the reactor (1.5 ft^3/hr at conditions) about 70% of the total product is vaporized. About 8% of the feed is converted to gases so that the liquid flow rate (density 1.0 g/cm^3) at the top of the reactor is only about 2.5 ℓ/h. For a 3.8-cm diameter reactor this represents a velocity of only 0.061 cm/sec* which is sufficient to fluidize and thus remove

*Stoke's law, fluid viscosity 0.23 cp, density difference 3.42 g/cm^3.

TABLE 1

Properties of Bitumen Feed Stock, GCOS 98

Specific Gravity	60/60^{o}F	1.010
Sulphur	wt %	4.73
Ash	wt %	0.56
Viscosity	cst at 210^{o}F	175.8
Conradson Carbon Residue	wt %	13.7
Pentane Insolubles	wt %	15.6
Benzene Insolubles	wt %	0.57
Nickel	ppm	68
Vanadium	ppm	211

Distillation Analysis

Equivalent Distillation
Range at 1 Atmosphere

Temp ^{o}C	Temp ^{o}F	wt %	Cumulative wt %	Sp Gr	Sulphur wt %
IBP-200	IBP-392	1.4	1.4	0.816	1.52
200-250	392-482	2.2	3.6	0.856	1.02
250-333	482-632	9.7	13.3	0.904	1.78
333-418	632-785	17.7	31.0	0.955	2.98
418-524	785-975	17.5	48.5	0.989	3.80
+524	+975	51.5	100.0	1.073	6.39

only very small particles (9 µm). An increase in the liquid-feed flow rate would decrease the conversion, which is not desirable, and a decrease in the recycle-gas flow rate would decrease the product vaporization with only small changes in the product composition. The best method of increasing the liquid flow rate at constant conversion is with heavy-oil recycle. With a maximum recycle pump flow of 14,380 cc/h the velocity would be 0.41 cm/sec, high enough to fluidize 23-µm particles. Another improvement in the reactor would be increased velocity near the wall which would decrease the boundary layer and perhaps reduce the volume of material lean in hydrogen. The amount of ash (natural catalyst) in the reactor liquid would also increase. In the hot separator the liquid velocity in the dip tube would be increased by a factor of about 6 to 7 and the turbulence in the bottom cone increased, thus preventing possible build-up of solid material. These improvements have to be weighed against the cost of operating another pump (the RO pump) at high temperature and pressure, and against changes in liquid yield.

The objectives of this work were to determine the operability of a thermal hydrocracking plant using a recycle-oil stream to increase liquid velocity in the reactor in an attempt to reduce coke or sludge formation and to determine the effect of heavy-oil recycle on product distribution and pitch conversion.

Of main concern in this report are those factors which influence the steady operation of the pilot plant such as coke formation leading to fouling and line plugging or deposition of metal and ammonium sulphide. The effects of heavy-oil recycle on pitch conversion, hydrogen consumption, sulphur, oxygen and nitrogen removal and on product distribution will be the subjects of another report.

APPARATUS

The apparatus was essentially the same as described in a previous report (8) except for modifications to allow for the heavy-oil recycle. The changes to the plant consisted of the addition of two 500-cm^3 capacity Ruska pumps for heavy-oil withdrawal, and a recycle-oil (RO) pump with nominal capacity of 3.8 U.S. gallons per hour for recycle of heavy oil.

A simplified flow sheet is shown in Figure 1.

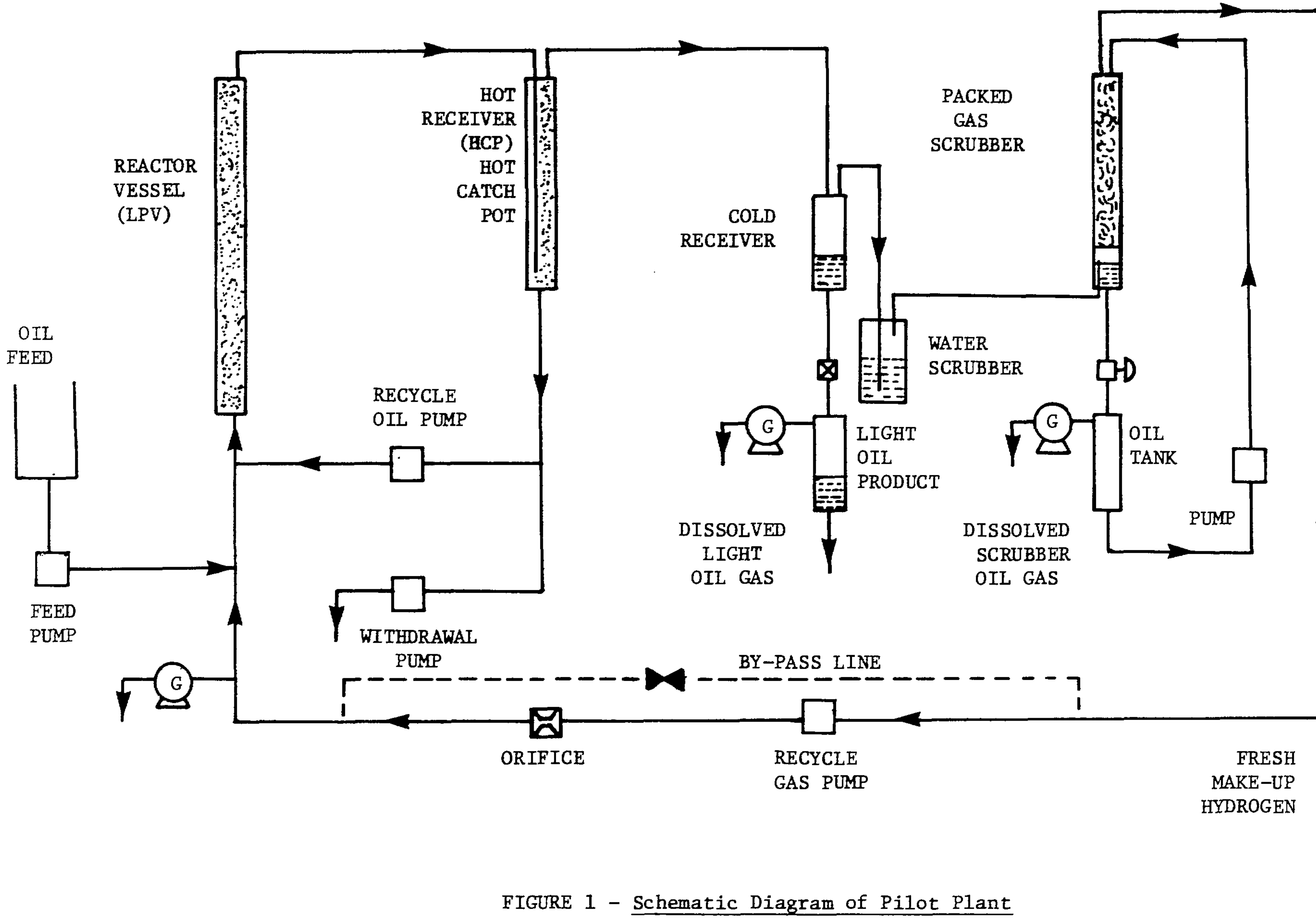

FIGURE 1 - Schematic Diagram of Pilot Plant

EXPERIMENTAL

1. Feed Stock

The feed stock used was bitumen separated from the Athabasca oil-sands deposits in northern Alberta. The properties of the bitumen (GCOS lot No. 98) and the results of a distillation analysis for this bitumen are shown in Table 1.

2. Operation

The heavy ends from the hot catch-pot (HCP) were recycled to the reactor bottom and a fraction was withdrawn (Figure 1). In the first few runs, the HCP level was maintained by adjusting the feed flow; in the later runs the HCP level was maintained by adjusting the RO withdrawal rate. At the start of this project, short runs were made at selected mild conditions (pressure = 2000 psi or 13.89 MPa, reactor temperature = 440°C, LHSV = 0.25 and RO withdrawal = 450 g/h). For long runs, the feed rate was increased to 4500 g/h (LHSV = 1.0) and the temperature was increased to 460°C. The run identification numbers and the conditions are given in Table 2.

At stabilized conditions, feed, liquid product and gas flows were carefully measured. For life runs, product samples for analysis were taken every day. Reactor-liquid samples were taken at three points of the reactor every fifth day.

3. Analytical Procedures

Analytical procedures were the same as described in the previous report (8).

TABLE 2

Reaction Conditions for Different Runs

Run No.	Feed Lot No.	Actual Feed Rate g/h	Nominal LHSV	Pressure psi	Reactor Temp °C	HCP Temp °C	Gas Flow ft³/h*	Gas Purity %	Actual RO Withdrawal Rate g/h	RO Pump Stroke in.	Run Time h	HCP Level Controlled by Adjusting:
R-1-1-1	98	2347	0.5	2000	440	440	1.5	85	459	1.5	4	Feed
R-1-2-1	98	2537	0.5	2000	450	400	1.5	85	489	1.5	23	Feed
R-2-1-1	98	4546	1.0	2000	450	450	1.5	85	1000	3.0	93	Feed
R-2-1-2	111	4535	1.0	2000	450	450	1.5	85	976	3.0	477	Feed
R-2-2-1	111	4492	1.0	2000	460	450	1.5	85	756	3.0	30	RO Withdrawal
R-2-2-2	111	4500	1.0	2000	460	450	1.5	85	≈1000	3.0	10	RO Withdrawal
R-2-2-3	111	4500	1.0	2000	460	450	1.5	85	≈1000	3.0	7	RO Withdrawal
R-2-2-4	111	4556	1.0	2000	460	450	1.5	85	748	3.0	283	RO Withdrawal

* At 2000 psi and 25°C

A detailed report on each run is given in the Appendix.

RESULTS AND DISCUSSION

In order to run a thermal hydrocracking plant smoothly for long periods of time, it is necessary to reduce the coke and sludge formation in the reactor or in the hot catch-pot (HCP). The criteria for smooth operation of the pilot plant were as follows:

1. smooth start-up
2. low and steady system pressure-drop
3. no coke deposition or sludge formation in reactor or hot separator
4. steady feed and product flows
5. steady hydrogen consumption
6. steady temperatures
7. no mechanical problems.

These criteria are identical to those of a full-scale plant and long runs will pinpoint problem areas. In this section of the report, the observations and problems encountered during the investigation will be discussed in the light of the above factors under the following subsections:

1. Preparation for the Run and the Modifications prior to the Run

Before the start-up of any run, except for run R-2-2-3 which was started immediately after the shutdown of run R-2-2-2, the pilot plant was cleaned thoroughly, especially the gas lines, and then flushed with varsol. Modifications to the plant were made for each run depending on the problems observed in the previous run.

The shutdown of run R-2-1-1 was due to a solid plug between the HCP and the light-ends receiver. The cause of the plugging may have been the carry-over of heavy ends to the light-ends section or the deposition of ammonium sulphide in the line. Although there was no definite proof (as no analysis of the material from the plugged line could be made for ammonium sulphide) the possibility of depositing ammonium sulphide did exist. The water traps installed before and after the recycle-gas compressors were packed nearly solid with ammonium sulphide and there were crystals in other lines and in the compressor check valves. To prevent further probelms with ammonium sulphide, a water scrubber was installed in the gas line just before the oil scrubber.

Heat balance calculations for run R-2-1-2 showed that the oil recycle rate was only 9060 g/h compared with 14,330 g/h rated RO-pump capacity. The computation showed that the recycle-oil-pump suction line (3.175-mm ID) was limiting, as the velocity in the 3.175-mm ID line was about 35.1 cm/s for 10,000g/h of total flow. For the next run the 3.175-mm ID line was replaced by a 4.76 mm line, reducing the velocity by a factor of 2.2.

The deviation in daily average feed rate for run R-2-1-2 was too high (4020 g/h to 4920 g/h) and the light-ends production and RO withdrawal rate followed the feed rate (Figure 2). The deviation in the feed rate could be due to adjustment of the feed rate to control the HCP level. Therefore, in the next runs the feed was kept constant at about 4500 g/h and the HCP level was maintained by adjusting the RO withdrawal rate, which proved to be satisfactory.

2. Start-up Procedure

For runs R-1-1-1 to R-2-1-1 the start-up procedure was the same. The liquid feed was pumped at a high rate when the reactor temperature was 350°C. When there was a sufficient amount of material in the HCP the feed was stopped and then the RO pump was started. The system was operated for about one hour on total recycle, after which the feed was started again at a lower rate to maintain the HCP level and the temperature of the reactor was increased slowly. The Ruska withdrawal pump was then set a a predetermined rate, to be maintained throughout the run. The procedure of constant withdrawal and variable feed rate to control the HCP level resulted in a wide variation in feed rates throughout the run, as shown in Figure 2 Accordingly, the procedure was modified for subsequent runs to have constant feed and recycle-oil withdrawal to maintain the HCP level during steady-state operation.

By trial and error through runs R-2-2-1 to R-2-2-4, the following start-up procedure was found to be successful:

1. heat the reactor to 350°C
2. start the feed and heat the reactor to 430°C
3. increase the temperature 5°C per hour after reaching 430°C
4. start recycling when the HCP level reached 30%
5. adjust feed rate to control the HCP level during start-up
6. make no withdrawal until reaction temperature reached
7. start steady feed rates and adjust withdrawal rates to control the level when at operating conditions

Run R-2-2-1 was started up with a steady feed rate and a varying RO withdrawal rate, but was shut down after 5 days because of electrical failure.

Run R-2-2-2 was started up in a similar fashion, but did not reach steady state: it seemed to plug in the gas lines because of carry-over of heavy ends. Finally run R-2-2-4 was started up as shown in steps 1 to 7 above, as it was throught this procedure would eliminate foaming of very heavy material in the initial stages of start-up. This procedure was successful.

3. System Pressure-Drop during the Run

For short periods the system pressure-drop is nearly constant and only for long runs (Figures 3 & 4) can changes in the system pressure-drop be seen. For run R-2-1-2 at 450°C (Figure 3) the system pressure-drop increased to 250 psi in about 20 days and the fluctuations in the total pressure-drop were high (up to 200 psi). For run R-2-2-4 (Figure 4) at 460°C the pressure drop steadily increased to 250 psi in about 12 days, but the fluctuations were less than in the run at the lower temperature, R-2-1-2. This could have been caused by better control of the HCP level effected by adjusting the RO withdrawal rate.

The system pressure-drop is a measure of plugging in the lines to the reactor, from the reactor to the hot catch-pot and in the gas lines. It is sometimes an indication of coke build-up in the reactor. It is usually

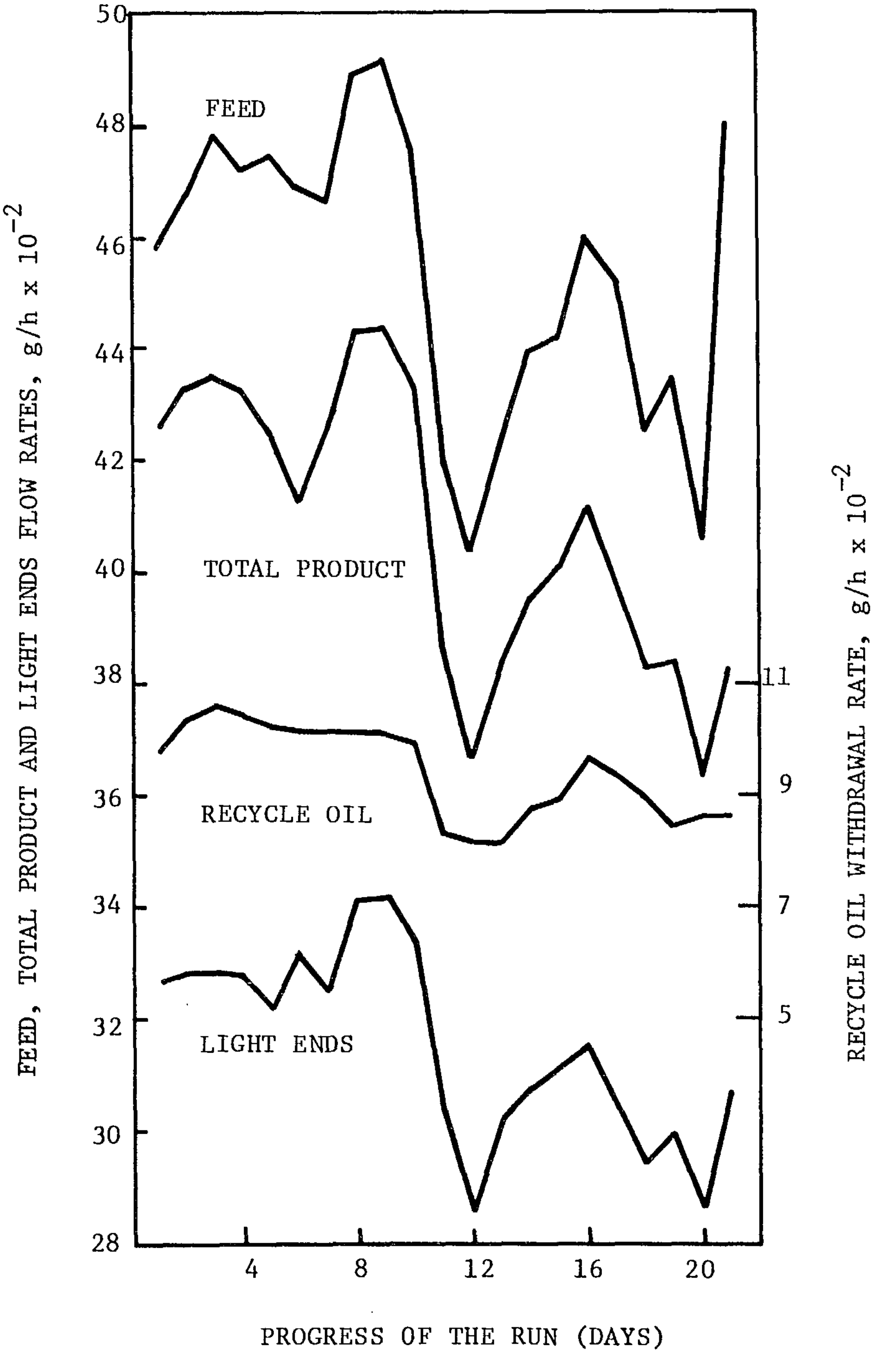

FIGURE 2 - Daily Average Feed and Liquid-Product Flows During Run R-2-1-2

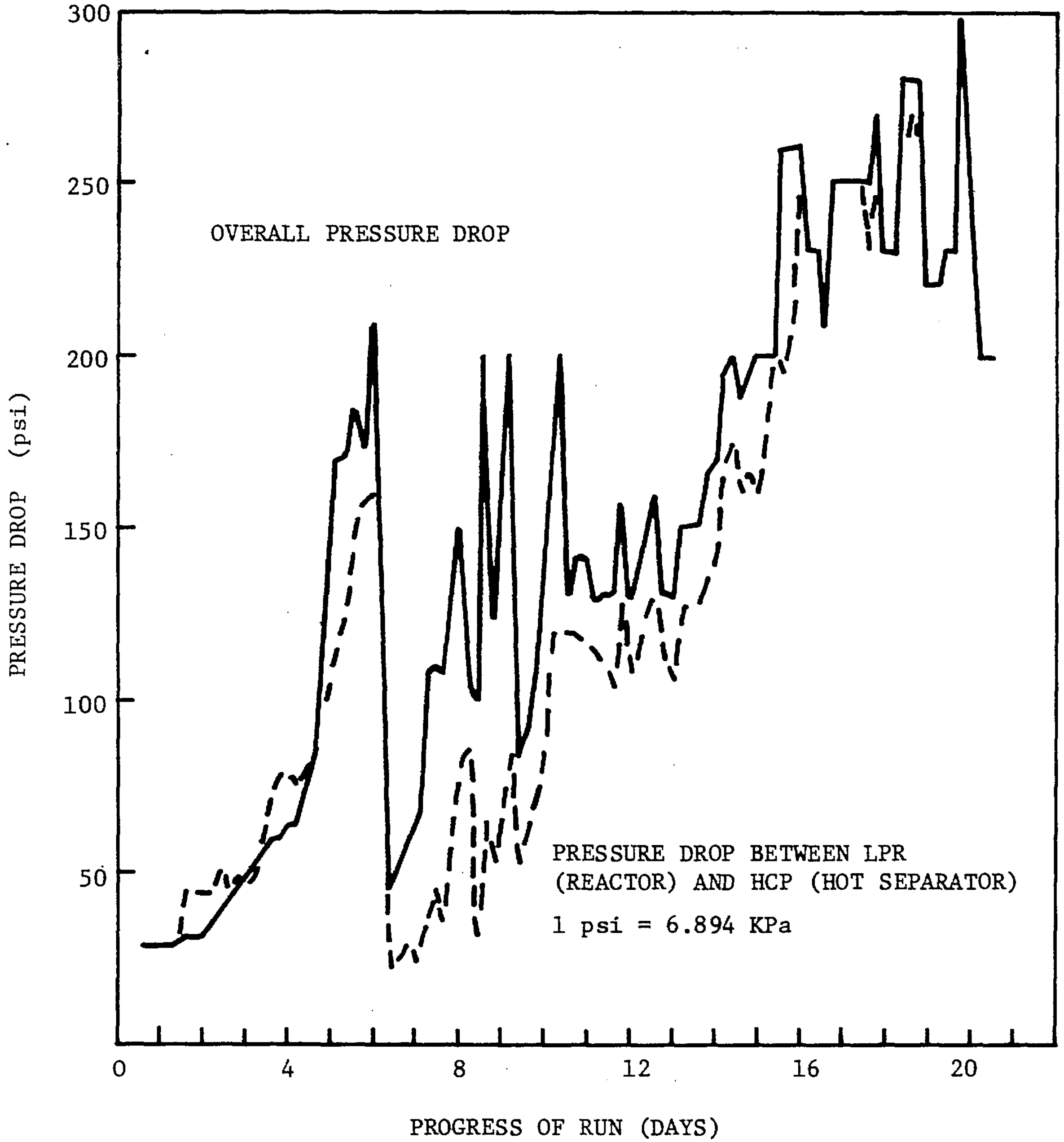

FIGURE 3 - Overall and LPR/HCP Pressure Drops during Run R-2-1-2

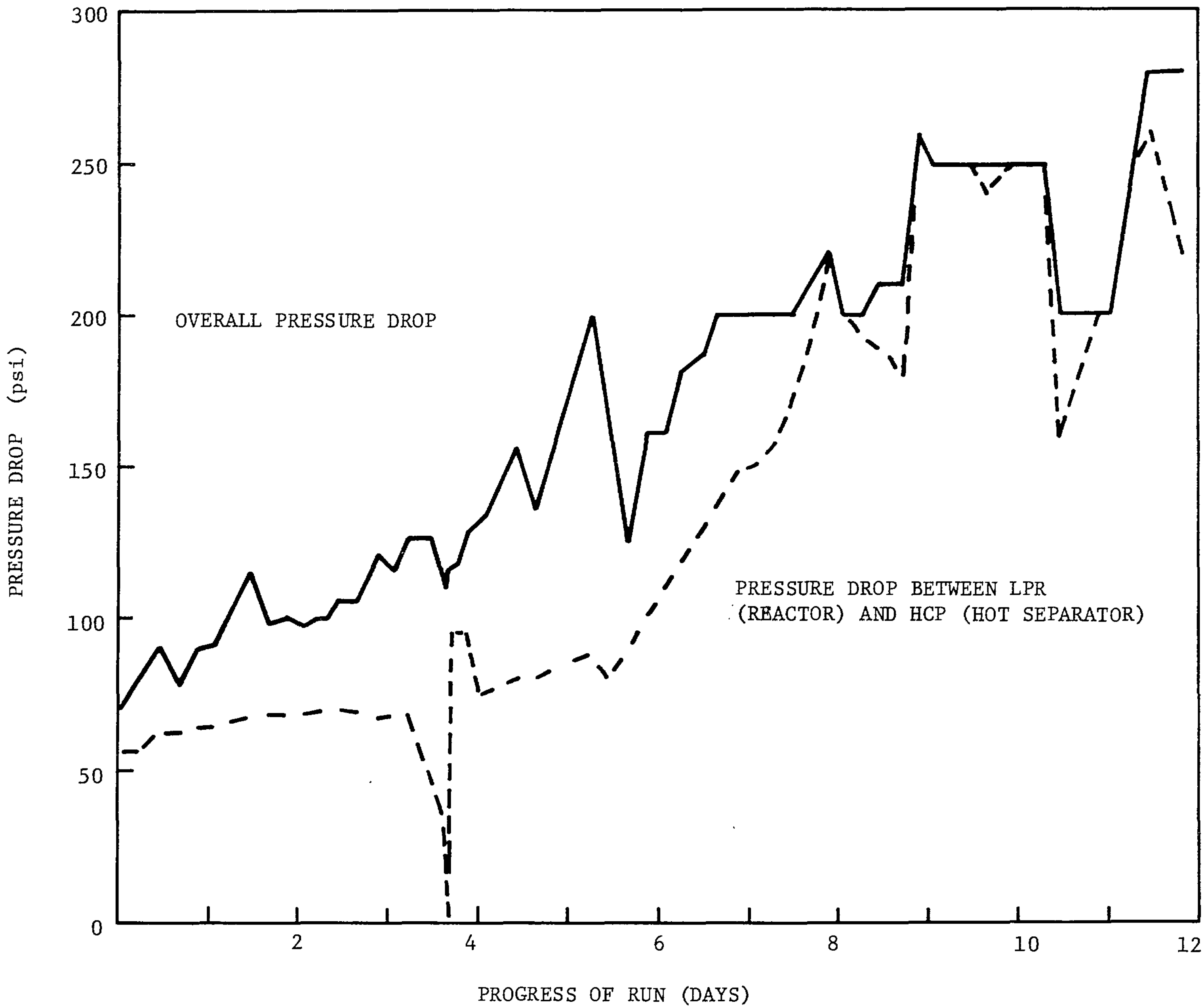

FIGURE 4 - Overall and LPR/HCP Pressure Drops During Run R-2-2-4

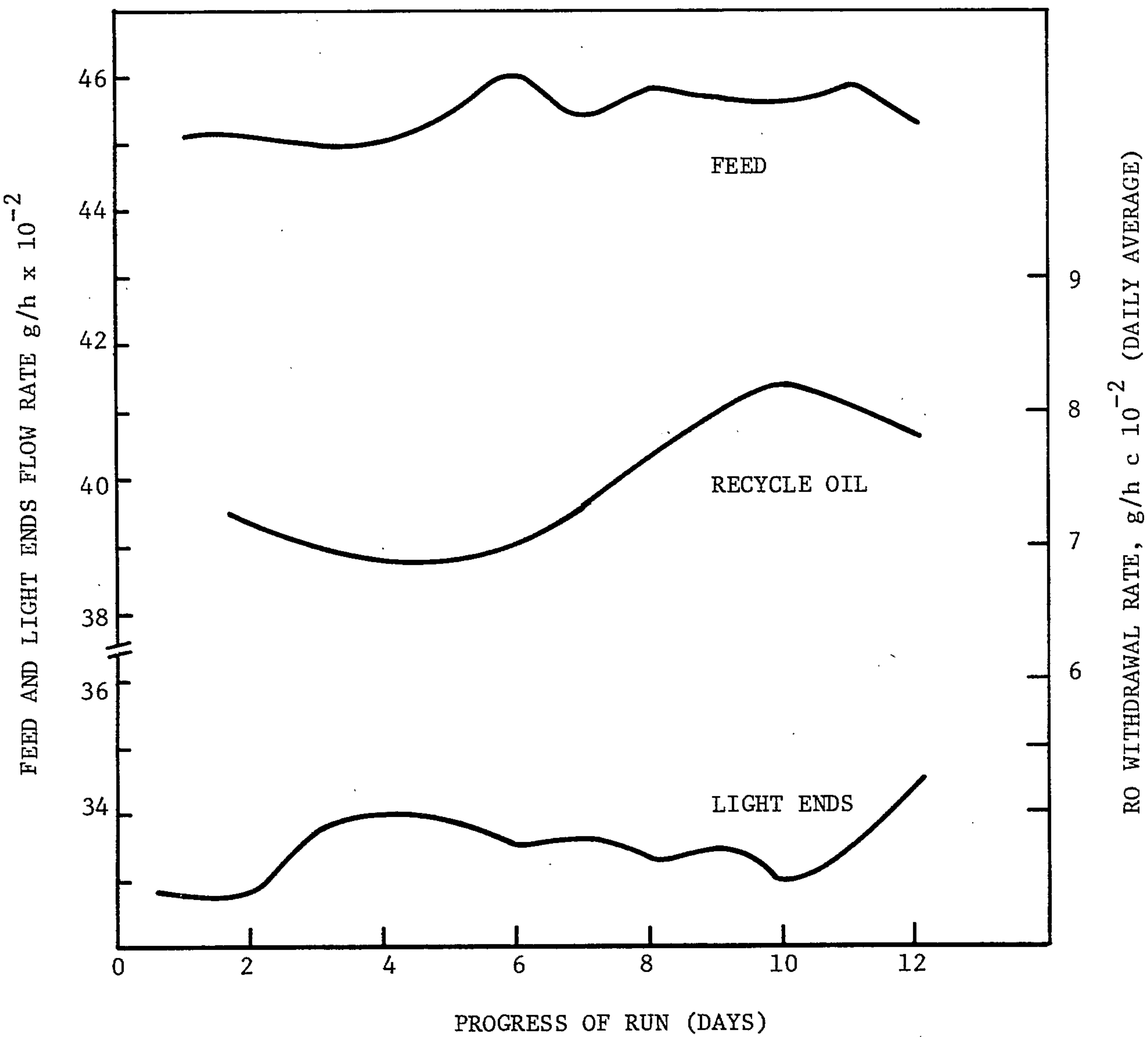

FIGURE 5 - Daily Average Feed and Liquid-Product Flows During Run R-2-2-4

caused by particles of ash from the feed, or by crystals of ammonium or iron sulphide in the transfer line. In the two recycle runs, R-2-1-2 and R-2-2-4, the pressure drop was relatively high and increased throughout the run. However the reactor was nearly free of coke deposits, with only a light dusting of coke on the walls.

4. Feed and Product Flows

For run R-2-1-1 the feed and liquid-product flows were nearly constant over its entire duration. The deviation in the daily-average feed rate for run R-2-1-2 (4020 to 4830 g/h) was too high (Figure 2). The variation in the feed rate may be due to its adjustment to control the HCP level. For run R-2-2-4 (Figure 5) the daily average feed rate was nearly constant over the total run which was expected because feed rate was kept constant. This shows clearly that to control the HCP level, adjusting RO withdrawal is much better than adjusting the feed flow rate. The light ends and recycle-oil production followed the feed flow-pattern (Figure 2). Feed flow rate affects the pitch conversion, hydrocarbon gas make, hydrogen consumption and sulphur removal. Fluctuations in feed rate cause fluctuations in reactor temperature and system pressure.

5. Gas Flows and Hydrogen Consumption

Gas flows were steady for all the runs. There were some fluctuations in recycle-gas flow for runs R-2-1-2 and R-2-2-4. These may be caused by problems with compressors and check valves. The daily-average hydrogen consumption was more for run R-2-2-4 (Figure 6) than for runs R-2-1-2 or R-2-1-1, but the fluctuations for run R-2-2-4 were less than for run R-2-1-2. This higher consumption of hydrogen was due to higher operating temperature leading to higher pitch conversion (85.4% for run R-2-2-4 and 81.5% for run R-2-1-2), higher sulphur removal (57.2 and 52.2% for runs R-2-2-4 and R-2-1-2 respectively) and higher hydrogen content in the light ends.

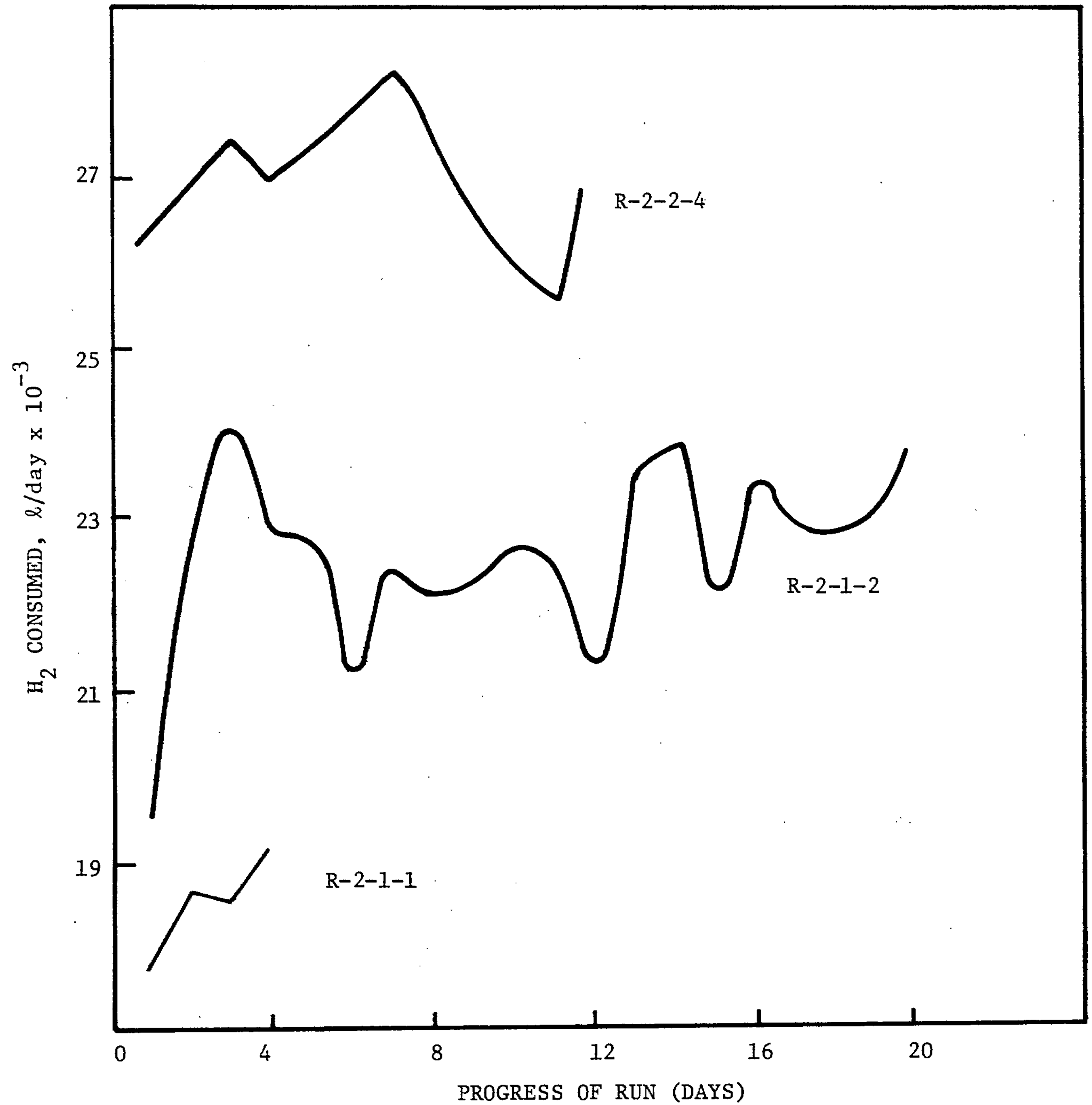

FIGURE 6 - Hydrogen Consumption

6. Pumps and Controls

For run R-2-1-2 the recycle-oil pumps began to leak at the pump packing during the run, with leaks as high as two litres over an eight-hour shift. This was probably caused by high pump-head temperature. For run R-2-2-4 there were problems with the gas recycle compressors. The main problem was failure of the inlet check valves due to carry over of "Drierite" water absorbent from the oil/water trap upstream from the compressors. Other problems included the lack of a facility for adding oil to the cylinder lubrication systems while running and defective (unsuitable material) compressor-inlet check-valve springs.

The packing on the oil-scrubber pumps was leaking throughout the run R-2-2-4. The oil scrubber removes hydrogen sulphide and hydrocarbon gases from the recycle gas, and gradual loss of oil results in poor operation.

7. Temperature of the System and Effect of Temperature

At all points of the system, temperatures were steady for all the runs except for run R-2-1-1 where all of the thermocouples in the RO section were defective.

There were no problems apparent in the plant operation for a short period at 440°C reactor temperature but when the temperature was raised to 450°C problems were encountered. The total system pressure-drop increased steadily to 250 psi in about 20 days. At 460°C, there were problems in the start-up of the run which were eliminated by improving start-up procedure but the total system pressure-drop increased to 250 psi in about 12 days. Coke formation in the reactor and the HCP was negligible even at 460°C reactor temperature.

8. Ash Concentration in the Reactor Liquid from Reactor Samples

The ash or mineral-matter content of the reactor liquid was expected to be constant at all points in the reactor because of the increased liquid-velocity which, at the top of the reactor, could be as high as ten times the velocity for a once-through operation at the same space velocity based on feed. Figure 7 gives a comparison between a once-through reaction (Run 84-1-1) and the recycle run at about the same conversion. The velocity at the top of the reactor for run R-2-1-2 was about 5 times the velocity

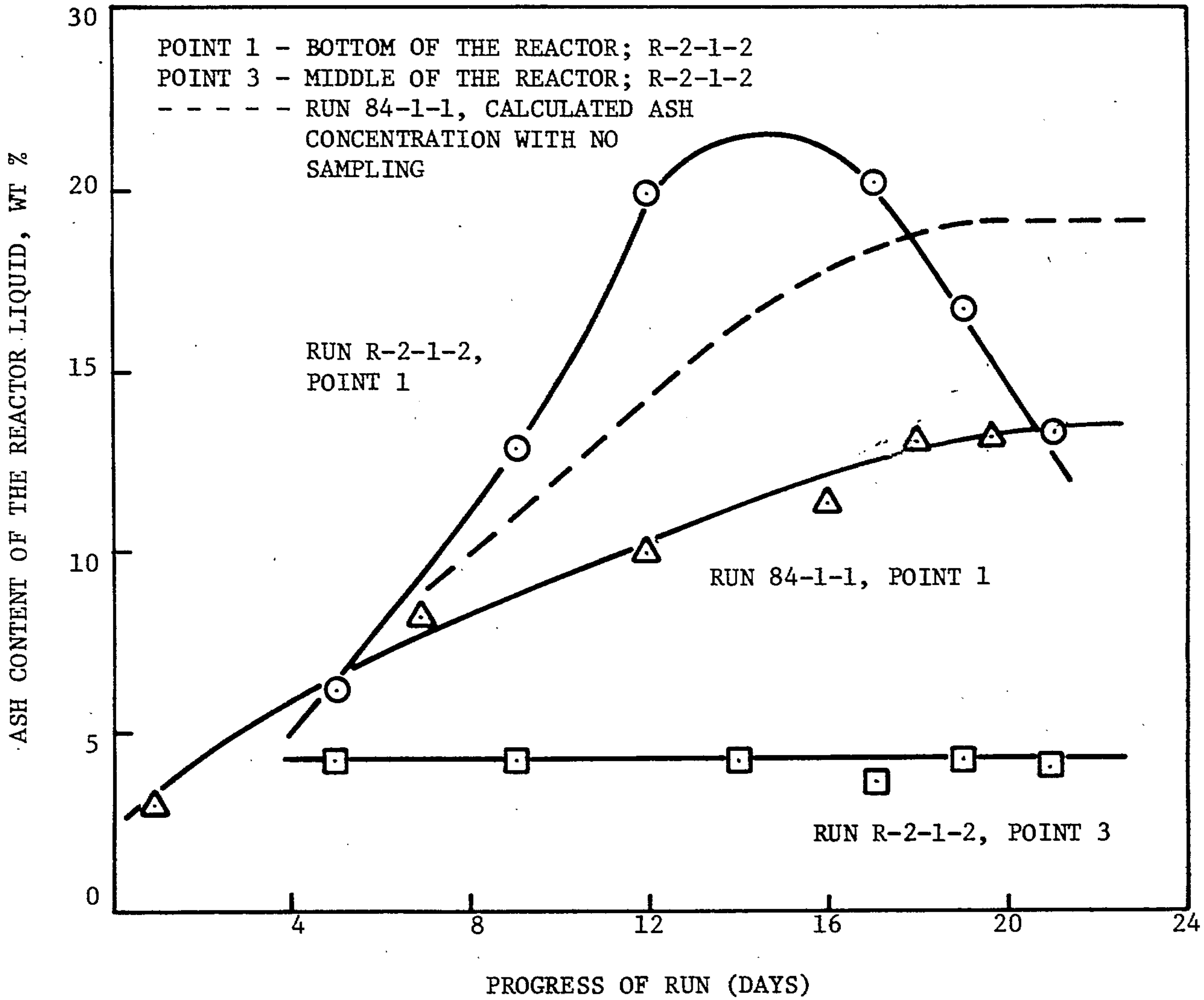

FIGURE 7 - Ash Content of the Reactor Liquid

run 84-1-1. The ash concentration at point 3 (middle of the reactor) for run 84-1-1, which is not shown, remained at 4% for almost the whole run, rising to 6% in the last three days.

One interesting point of comparison for the two runs is that the input of ash or mineral matter for the once-through run was double that of the recycle run. Furthermore, the ash input for the recycle run was sufficient to fill the reactor with ash to the 20% level in less than 20 hours if all ash were retained.

The dotted line in Figure 7 is an estimate of the true reactor ash-content in run 84-1-1, corrected for the ash removal during sampling.

9. Inspection after Completion of the Run

The plant was disassembled after each run with the exception of run R-2-2-2. The reactor, hot catch pot, light-ends receiver, water and oil scrubber and all the lines were checked for coke deposition or any type of contamination in the system. For each run a detailed report of this inspection is given in the appendix. For all runs, the system was checked part by part.

(a) Reactor outlet cap - The reactor cap was always clean except after the long runs, R-2-1-2 and R-2-2-4. For run R-2-1-2 the cap had a hard foam-like material and for run R-2-2-4 it had a small amount of bubbly oil.

(b) Reactor wall - Generally the reactor was clean except after runs R-2-1-2 and R-2-2-4. For run R-2-1-2 there was a light dusting of powdery material with an occasional "boil" about 1 cm in circumference. For run R-2-2-4 the reactor wall and the thermocouples were coated with black powder. The coating was thickest within one metre of the reactor top.

(c) Reactor bottom cap - The bottom cap always contained some black powder. In run R-2-1-2 there was black powder and a small amount of oil.

(d) HCP - For run R-2-1-2 the lower portion of the hot receiver had a black powdery material caked as much as 2 mm thick on the walls. whereas for run R-2-2-4 the lower 1-m portion had a very thin layer (<1 mm). Except for the lower portion of the hot receiver the vessel was clean. The outlet cap had a very small amount of oil for run R-2-2-4 and it was full of porous black material for run R-2-1-2. For other runs it was clean. The down tube and the thermowell had a 2- to 3-mm thick coke deposit for all the runs.

(e) Light-ends receiver - For all the runs the light-ends receiver had some carry-over from the HCP, about 200 g of black material found to be heavy-oil product.

(f) Traps before compressor - For long runs, the dessicant was black at the trap exit and yellow-gray at the inlet.

(g) Down pipe in the HCP - For the long runs the down pipe was coated with a 2- to 3-mm thick black material but the holes of the sparger were open.

(h) Preheaters - The preheater section of the line was clean (except for some solid for run R-2-1-2).

(i) Other parts - Except for run R-2-2-4, other parts of the system were clean. The left piston of the oil scrubber pump was badly scored. Small amounts of the heavy oil were in the gas-recycle line.

10. General Discussion and Comparison with Once-through Runs

There were no problems of coke deposition or fouling of the reactor or the HCP for the short, once-through runs at 2000 psi and 450 and 460°C. Increase in the operation time, however, caused coke deposition in either the reactor or the HCP. Fouling of the reactor or the HCP made it extremely difficult to operate the plant for longer periods of time under certain conditions. Hence no life runs were made at LHSV = 1.0, P = 2000 psi and a temperature of 460°C. Accordingly no direct comparison can be made between long once-through and the long heavy-oil recycle runs. However, the coke formation in the reactor or the HCP was considerably suppressed by recirculating the heavy ends. The suppression of the coke formation may be due either to the increased liquid flow in the reactor or to the increased ash content of the reactor fluid. With the recirculation of heavy ends, the plant could be operated smoothly for a longer period of time without plugging the reactor or the HCP.

The start-up procedure was smooth with total heavy-ends recycle before the desired temperature was reached. The feed and liquid-product flows were made steady by adjusting the RO flow to maintain the HCP level. There were not many problems with the pumps and check valves except for run R-2-2-4. These problems were avoided with the modifications suggested in the recommendation sections in the Appendix.

The usual cause of shutdown was that a plug developed in the line, especially in the light-ends section. It is clear that heavy-oil carry-over to the light-ends section was the rule rather than the exception. The five-day run R-2-1-1 was shut down because of heavy-oil carry-over. The 20-day run R-2-1-2 was successful, but the clean-up revealed some heavy-oil carry over in the light-ends receiver. Run R-2-2-2 was shut down possibly because of carry-over and run R-2-2-3 was definitely shut down because of carry-over from either foaming or high HCP level. For further runs with recycle, the plant needs some modifications to avoid this heavy-oil carry-over to the light-ends section.

In runs where the feed passes through on a once-through basis, the residence time can be considerably longer than the reciprocal of the space velocity. This is because there is vaporization of the feed and product liquids. In the recycle runs the average residence time for the first feed pass (0.3 h) is much shorter than for a single pass or once-through operation (1.5 h). This is based on a gas holdup of 25%, a liquid density of 1.0 g/cm^3, a 90% liquid yield, and 70% and 30% product and feed vaporization respectively. A recycle flow of 9060 g/h and LHSV = 1.0 based on feed and RO density of 1.0 were also assumed. However, the total residence time of the heavy oil can be considerably longer with the system liquid-volume/heavy-oil withdrawal ratio of (4500 x 0.75 + 300)/1000 ≈ 3.7 h, based on a HCP liquid volume of 300 cm^3, heavy-oil withdrawal of 1000 g/h and heavy-oil density of 1.0 (extrapolated from Fig 77, Ref 10). In other words, the average residence time of the heavy oil is 3.7 hours although some can be in the system for one pass (0.3 h) and theoretically some can be in for the whole run. This work has shown that it is the first pass which is important, and that the heavy oil which can stay in the system for an average of about 3.7 hours appears to be more stable with respect to coke formation. Alternatively, the additional liquid can be scouring the walls, although this hypothesis is difficult to believe because the liquid velocities are small - in the order of less than one cm per second - even with recycling.

The solids in the reactor were concentrated as much as 40 times more than in the feed. The accumulation of solids in the reactor may be due to the fact that sand particles in the feed have a wide particle-size distribution ranging from less than 5 microns to more than 50 microns (1) There is a tendency for the larger particles to remain in the reactor because of gravity, if the flow force is not sufficient to take them out of the reactor, while the smaller ones are carried out. Alternatively there may be some tendency for particles to agglomerate due to adsorption of larger oil molecules which tend to be sticky. Because of the upflow of the liquid and the mixing action of the gases, mineral-matter particles will be in a state of random motion. A mass of solid particles in this state of random motion in a liquid medium may be described as ebullated. The random motion of particles in the ebullated mass causes these particles to rub against each other and against the walls of the vessel so that the formation of deposits is minimized. This scouring action helps to prevent agglomeration of the particles and plugging of the vessel. Although the fluid velocity in the reactor was small (0.28 cm/s average for run R-2-1-2 and 0.36 cm/s average for run R-2-2-4 where the recycle flow was increased to 12,700 g/h) it should have been sufficient to put small particles in random motion.

Chervenak et al.(1) observed that a feed with high ash content is less likely to deposit coke in the reactor and that hydrogen consumption increases with increasing amounts of ash in the reactor fluid. They have also stated that, if the hydrogen content of the heavy-oil product is more than 6.8 wt %, no coke will deposit in the vessel. In the present investigation, the ash content of the reactor fluid increased to about 20 wt %. The total recycle of heavy ends during the six-hour start-up period, i.e., until the desired temperature was reached, yielded an ash content in the reactor fluid of 3 wt %. According to Chevernak et al. (1) this should have been sufficient to prevent fouling of the reactor. Hence the start-up procedure for run R-2-2-4 was smooth. The increasing amount of ash in the reactor fluid gives a higher hydrogen consumption which contributes to an increase in the hydrogen content of the light ends, the recycle oil or the pitch. For run R-2-2-4 the hydrogen content of the pitch was about 6 wt % which is not considered to be sufficient to inhibit fouling of the hot receiver. But even with this hydrogen content of less than 6.8 wt % in the pitch, there was no coke deposition in the reactor or in the hot catch-pot, contrary to the claim of Chervenak et al. (1). The total recycle of heavy ends in the start-up is an improvement over the process involving addition of an appropriate hydrogenation catalyst in the initial stages of the run.

Wolk et al. (2) have plotted temperature in the separator (linear scale) against the liquid velocity in the separator (log scale). They have drawn a curve to form a demarcation zone between two areas as shown in Figure 8. Area A indicates coke-free conditions and area B generally indicates coke formation. Wolk et al. (2) also stated that, to prevent coke formation, the temperature of the separator should be between 500 and 800^{o}F (260-427^{o}C) and the liquid velocity in the separator should be 0.006 - 1.0 ft/sec (0.18 - 30 cm/sec). The lower velocity corresponds to the lower temperature. In the present investigation, the temperature in the separator was 450^{o}C and the liquid velocity was 0.24 cm/sec for run R-2-1-2 and 0.34 cm/sec for run R-2-2-4. According to Wolk et al. (2), these conditions should result in the deposition of coke in the separator. However, in the present investigation, except for slight dust in the separator (HCP) or a small amount of coke on the dip tube, no major problem or fouling of the hot receiver was encountered. This indicates that with the heavy-oil recycle the plant could be operated without fouling of the separator in the coke formation zone as suggested by Wolk et al. (2).

Wolk (4) has shown also that, by passing the heavy ends through a liquid cyclone or a low-pressure separator and recirculating the ash-enriched underflow at a recycle ratio of 0.05 to 1.0 (ratio of ash-enriched fraction to fresh feed), thus maintaining the ash content of the reactor fluid between 4 and 10 wt %, the plant could be operated without fouling of the reactor at a conversion between 75 wt % (t = 449^{o}C and recycle ratio = 0.001) and 87.7 wt % (t = 454^{o}C and recycle ratio = 0.9). In the present investigation the underflow of the hot receiver was recycled without further treatment, i.e., without using a liquid cyclone or a low-pressure separator. The recycle-oil/feed ratios were 2.0 for run R-2-1-2 and almost 3.0 for run R-2-2-4. The plant was operated for a long period of time without fouling of the reactor or the hot receiver, with a conversion (disappearance of +975^{o}F fraction) of about 82-86 wt %. This was clearly contrary to previous results and is an area which merits further study.

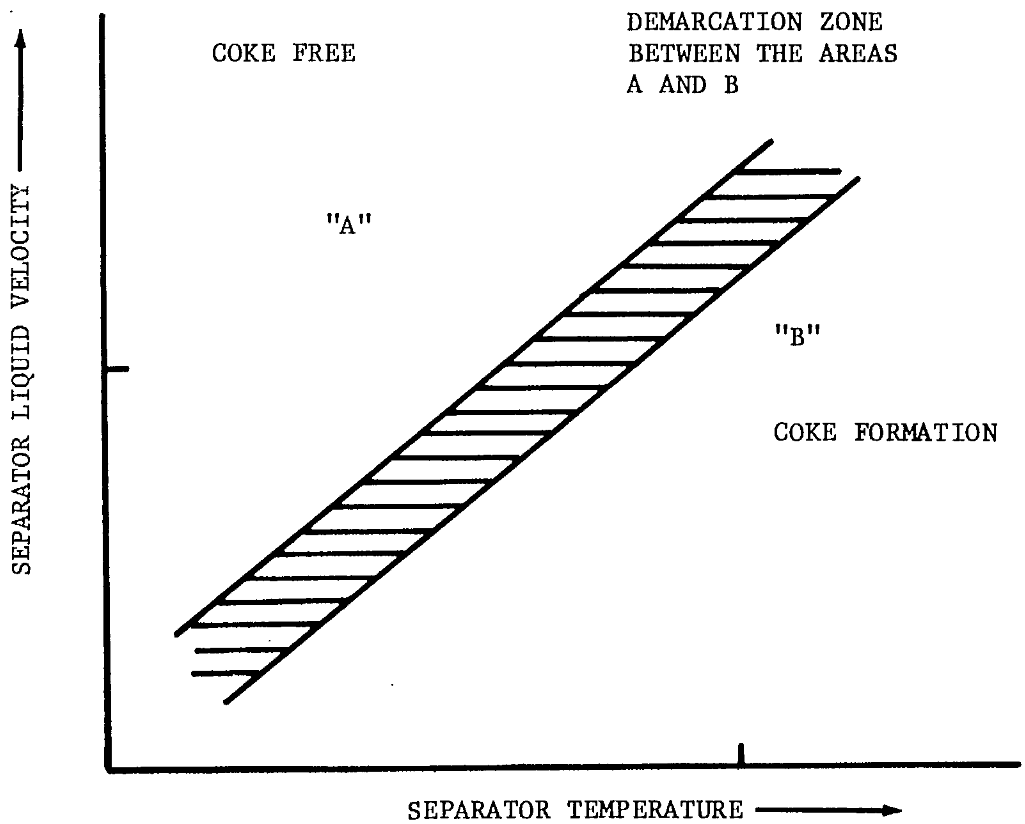

FIGURE 8 - Effect of the Separator Liquid Velocity and Separator Temperature on Coke Formation (Ref. 2)

CONCLUSIONS

Coke and sludge formation in the reactor and in the hot receiver could be suppressed significantly by recycling a fraction of the heavy ends to improve the operability of the plant for hydrotreating Athabasca bitumen. The plant could thus be operated for longer periods of time without fouling of the reactor or the HCP.

Total recycle of the heavy ends before reaching the required temperature gave a smooth start-up. Adjustment of RO-withdrawal rate was better than adjustment of the feed flow for maintaining the hot-separator (HCP) level. This gave smooth liquid-flow rates and fewer fluctuations in the total system pressure-drop.

Comparison of our results with those revealed in certain patents indicates inconsistencies, and even applications of their general conclusions were apparently invalid. This comparison merits further study.

Certainly the success or failure of a run depends on the ash content of the reactor liquid and recycling of heavy oil tended to increase this reactor-ash content over the single-pass operation. This increase in ash content, coupled with a short "first-pass" residence time were perhaps the reasons for the success of the recycle operation.

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This work was initiated by W.H. Merrill, who for many years has been a proponent of recycling to improve plant operation.

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APPENDIX A

Detailed Reports on Individual Runs.

July, 1974 RUN NO. R-1-1-1 4 hours

Conditions

Feed	GCOS Bitumen, Lot No. 98	
Feed rate	2200 g/h	LHSV 0.5
Pressure	2000 psi	
Reactor	13 ft x 1½ in. inside dimensions	
Temperatures		
	Reactor	440°C
	HCP	440°C
Gas flow	1.5 cfh	
Purity	85%	
RO withdrawal	459 g/h	
RO pump stroke	1.5 in.	

Purpose

The purpose of this run was to explore the possibility of heavy-oil recycle, to determine operational problems that might be encountered and to test for gross equipment malfunctions for the hydrocracking of Athabasca bitumen.

Changes since Previous Run

This was the first run with heavy-oil recycle.

Preparation for Run

General clean-up.

Start-up

The reactor was heated to 350°C and the feed was pumped at a high rate. When a sufficient amount of material was in the HCP, the feed flow was stopped and the RO pump was started. After operating the plant for about one hour on total recycle, the feed flow was started again at a lower rate. The temperature of the reactor was adjusted to the desired level, the RO withdrawal started and the feed rate was adjusted to maintain the HCP level.

Operations

Pressure drop - The pressure drop across the system was nearly constant (72 psi) throughout the run.

Feed and product flows - Feed flow varied between 2180 and 2580 g/h with an average value of 2348 g/h. Light-ends production (1825 to 1474 g/h) followed the feed flow. RO production was nearly constant.

Gas flows - Gas flows and hydrogen consumption were steady.

Temperatures - All the temperatures were steady during the run.

Pumps and controls - Pump operations were good.

Cause of Shutdown

Completion of the run.

Inspection after Completion of Run

Interviews with personnel indicated that the system was clean.

Discussion

This run was not long enough for true steady state or for operational problems to be encountered.

Conclusion

The data for a very low effective-space velocity (0.4 h^{-1}) should be interesting for comparison purposes.

Recommendations

1. The next run should be operated for a longer period of time.
2. Temperature of the reactor should be increased to 450°C.

July 10-11, 1974 | RUN NO. R-1-2-1 | 23 hours

Conditions

Feed	GCOS Bitumen, Lot No. 98	
Feed rate	2500 g/h	LHSV 0.5
Pressure	2000 psi	
Reactor	13 ft x 1½ in. inside dimensions	
Temperatures		
	Reactor	450°C
	HCP	440°C
Gas flow	1.5 cfh	
Purity	85%	
RO withdrawal	500 g/h	
RO pump stroke	1.5 in.	

Purpose

The purpose of the run was to check the operability of the plant for a longer period of time.

Changes since Previous Run

Reactor temperature = 450°C

Preparation for Run

General clean-up.

Start-up

Same as for run R-1-1-1

Operations

Pressure drop - System pressure drop was steady at 70 psi over the run.

Feed and product flows - Feed flow was nearly equal to 2600 g/h. Product flows were steady over this period. There were problems with the recycle-oil flow prior to the run starting, but no problems during the 24 hour period.

Gas flows - Gas flows were steady during the run.

Temperatures - Temperatures were steady.

Operations (Cont'd)

Pumps and Compressors - The Ruska (RO-withdrawal) pumps and the RO pumps were found to be satisfactory.

Cause of Shutdown

Completion of the run.

Inspection after Completion of Run

Interviews with personnel indicated that the system was clean.

Discussion

There were no operating problems during the run. Data was collected for a very low (0.5 h^{-1}) effective-space velocity. It is unlikely that further runs will be made under these conditions.

Conclusion

The plant operation was normal at a lower space-velocity (0.5 h^{-1}).

Recommendation

As there were no problems in the operation of the plant using a recycle-oil stream at an effective-space velocity of 0.5 h^{-1} and 450^{o}C, it seems that the plant could be operated at higher space-velocities and temperatures. Hence further studies should be made at LHSV = 1.0 and reactor temperatures of 450 and 460^{o}C.

January 13-17, 1975 RUN NO. R-2-1-1 93 hours

Conditions

Feed	GCOS Bitumen, Lot No. 98	
Feed rate	4500 g/h to control HCP level	LHSV = 1.0
Pressure	2000 psi	
Reactor	13 ft x 1½ in. inside dimensions	
Temperatures		
	Reactor	$450^{o}C$
	HCP	$450^{o}C$
Gas flow	1.5 cfh	
Purity	85%	
RO withdrawal	1000 g/h	
RO pump stroke	3.0 in.	

Purpose

To determine operability of thermal-hydrocracking plant using a recycle-oil stream to increase liquid velocity in the reactor in an attempt to reduce sludge or coke formation.

Changes since Previous Run

1. LHSV = 1.0
2. RO pump stroke = 3.0 in. (max of 1 side)
3. RO-withdrawal rate = 1 litre/hr = 1000 g/h

Preparation for Run

General clean-up.

Start-up

Same as for run R-1-1-1.

Operations

Pressure drop - After 1 day, pressure drop increased steadily until it reached about 100 psi. This ΔP was not localized; each gauge showed a slight increase from No. 1 to 4. On Friday, Jan. 17th at 8:10 P.M.

a plug developed, then cleared and, in so doing, some of the reactor contents were thrown over to the HCP. Some of the HCP contents were carried over in the gas line and to the LE receiver, plugging the line solidly. Pressure went up to 4200 psi on gauge No. 3.

Feed and product flows - Feed flow varied between 3630 and 5210 g/h with an average value of 4546 g/h. Daily-average value of feed flow increased slightly every day for the whole run, as it did for light ends and recycle oil.

Gas flows - Gas flows were steady. Hydrogen consumption increased with the progress of the run.

Temperatures - Temperatures were steady except for the RO section. Thermocouples in the RO system did not seem to be recording properly. A check revealed that 4 of them were defective.

Pumps and controls - After 1 day, the RO pump (left side) ceased to operate. Operation was switched to the right side. However, after check valves were flushed, the left side pumped well for a 15-min check. Other pumps operated well.

Cause of Shutdown

A solid plug developed between the HCP and the LE receiver.

Inspection after Completion of Run

Reactor - The outlet reactor-cap and the inlet reactor-cap were clean. Reactor walls had a light dusting. 4-5 tablespoons of powdered coke were removed from the reactor vessel.

HCP - The hot catch pot was clean except for a fine fuzz on the walls of the vessel.

LE receiver - It was clean.

Traps before the gas compressor - They were clean.

Preheaters - The line was clean.

Down-pipe in the HCP - The dip tube in the HCP was clean except for a fine fuzz on the thermowell.

Discussion

For a five-day period the plant operated well. The cause of the plug could be carry-over from the HCP or, more probably, ammonium sulphide deposited in the line. No coke or sludge formed in the reactor or the HCP.

Conclusion

The plant could be operated for a longer period of time provided plug formation in the LE section or HCP carry-over could be avoided.

Recommendations

1. There should be a temperature control on the light-ends cooler and receiver.
2. A water scrubber should be installed in the gas line before the oil scrubber to remove ammonium sulphide from the gas stream.

Feb. 23 - March 14, 1976 RUN NO. R-2-1-2 477 hours

Conditions

Feed	GCOS Bitumen, Lot No. 111	
Feed rate	4500 g/h to control HCP level	LHSV = 1.0
Pressure	2000 psi	
Reactor	13 ft x $1\frac{1}{2}$ in. inside dimensions	
Temperature		
	Reactor	450°C
	HCP	450°C
Gas flow	1.5 cfh	
Purity	85%	
RO withdrawal	1000 g/h	
RO pump stroke	3 in.	
RO recycle rate	9060 g/h	

Purpose

To determine operability of thermal-hydrocracking plant using a recycle-oil stream to increase liquid velocity in the reactor in an attempt to reduce sludge or coke formation.

Changes since Previous Run

1. Better control of light-ends-receiver temperature
2. Water scrubber installed before oil scrubber to remove ammonium sulphide from the gas stream.

Preparation for Run

General clean-up.

Start-up

Same as for run R-1-1-1.

Operations

Pressure drop - The pressure drop across the system increased steadily over the 20-day run and it was clear that the run could not have continued much longer at the time that the normal shutdown was instituted.

Feed and product flows - The feed flow varied from a high of 4900 g/h to a low of 4050 g/h with an average of about 4400 g/h. Light-ends production followed the feed flow, as did the actual RO withdrawal. It was found that the RO withdrawal rate from the Ruska pumps depended on which end of the recycle-oil pump was being used. This pointed out the possibility that the suction lines to the RO and Ruska pumps were limiting the flow. On checking the RO flow by heat balance, it was found that the flow was only 9060 g/h for the full stroke compared to 14,380 g/h rated RO pump capacity (3.8 US gph). This data and the other observations led to the conclusion that the suction was limited. (Computations showed that the velocity in the 3.175-mm ID line was about 33 cm/s for 10,000 g/h total flow.)

Based on the above, a decision was made to increase the size of the line and of the outlet of the hot catch-pot (HCP) to 4.76-mm ID, a total of 11 ft (3.35 m) of line, prior to the next run.

Gas flows - Hydrogen consumption was steady during the run.

Temperatures - Temperatures were steady during the run, with an occasional 5°C upset in preheater temperatures due to running empty in the HCP.

Pumps and compressors - The recycle-oil pumps, which were used in turn, both began to leak through the pump packing during the run, with leaks as large as two litres during an eight-hour shift. This was probably caused by high pump-head temperatures, which were about 360°C for the first five days and 410-415°C for the remainder of the run.

Feed-pump operation was good. Recycle-gas compressor operation was good, with some problems due to dirty inlet check-valves.

Cause of Shutdown

Completion of the run.

Inspection after Completion of Run

Reactor - The reaction vessel had a light dusting of powdery material on the walls, with an occasional "boil" about one cm in circumference. The outlet reactor-cap had a hard foam-like deposit through which the total flow had to pass. The inlet reactor-cap had a light dusting of powdery material.

Hot receiver (hot catch pot)- The hot receiver had black powdery material caked on the walls as much as 2-mm thick in the lower one-meter portion. The outlet reactor-cap was full of porous black solid material, and the internals (dip tube and thermocouple) had black material 2- to 3-mm thick caked on the lower portion, up to about one meter, with a thinner coating above this height.

Light ends receiver - This had some carry-over from the HCP which had become impregnated with a metal sulphide and H_2S. The material was black and "gooey".

Gas lines - All the gas lines were clean.

Traps before gas compressor - These had a light dusting of grayish powder in them. The oil reservoir was nearly empty, with only 2 inches of oil left in the bottom.

Preheaters - All three were caked with material, restricting the area through which the flow had to pass by one third.

Down pipe in HCP - This was very heavily caked with hard black material. The holes in the sparger were plugged.

Other problems - A reactor heater (second from the bottom) burned out and shorted.

Discussion

This was a successful run but variations in the total system pressure drop, feed rate, LE and RO production were high. The LE and total liquid-product flow rate followed the feed rate. These fluctuations could be due to the adjustment of the feed to control the HCP level. The HCP level could also be controlled by adjusting the RO withdrawal rate. The control of the HCP level by adjusting the RO withdrawal rate and keeping the feed rate constant could avoid these fluctuations in the liquid flow.

Conclusion

The plant could be operated under run R-2-1-2 conditions for about twenty days, but not much longer. The carry-over from the hot receiver to the light-ends receiver points to either a high level in the hot receiver, or foaming, or both.

Operation was much improved over runs at 450°C, 2000 psi and space velocity 1.0 without recycle, which could not continue for more than 16 days (Run 77-1-1 was shut down in 16 days). This run had a temperature of 450°C, pressure of 2000 psi and a space velocity of 1.5.

Recommendations

1. The HCP level should be controlled by adjusting the RO withdrawal rate and the feed rate should be kept constant.
2. The reactor temperature should be changed to 460°C.
3. The HCP outlet line (3.175-mm ID) should be changed to 4.76-mm ID.

March 24-25, 1976 RUN NO. R-2-2-1 30 hours

Conditions

Feed	GCOS Bitumen, Lot No. 111	
Feed rate	4500 g/h	LHSV ≈ 1.0
Pressure	2000 psi	
Reactor	13 ft x 1½ in. inside dimensions	
Temperatures		
	Reactor	460°C
	HCP	450°C
Gas flow	1.5 cfh	
Purity	85%	
RO withdrawal	≈850 g/h to control the HCP level	
RO pump stroke	3.0 in.	

Purpose

The purpose of this run was (1) to determine the operability of the thermal-hydrocracking plant using a recycle oil in the reactor stream, (2) to check the effect of RO withdrawal rate to control the HCP level and (3) to determine the effect of increased temperature (460°C) on plant operability, product distribution and pitch conversion.

Changes since Previous Run

1. Reactor temperature ≈ 460°C
2. Feed rate ≃ 4500 g/h but constant
3. Variation in the RO withdrawal rate to control the HCP level.

Preparation for Run

1. General clean-up.
2. Feed and RO pump-heads were re-packed.
3. A bypass was installed on the water scrubber.
4. Existing 3.175-mm ID line from the HCP to the RO pump was replaced with a 4.76-mm ID line. Approximately two meters of line were replaced, and the HCP outlet was increased in size to 4.76 mm.

Start-up

The reactor was heated to 350°C. The system was filled with feed. When a sufficient amount of feed was in the HCP, the feed pump was stopped and then the RO pump was started. The temperature was increased rapidly to 430°C and the feed pump was started again at a rate of 4500 g/h. The RO withdrawal-rate was adjusted in order to maintain the HCP level. The temperature of the reactor was increased slowly (5°C/h). After 6 hours, the temperature of the reactor was 460°C.

Operations

Pressure drop - The system pressure-drop increased from 50 psi to 80 psi over the run period. This was probably caused by an improved RO flow.

Feed and product flows - Feed flow was steady. Control of the HCP level by RO withdrawal appeared to be satisfactory. There were some problems with RO withdrawal and the RO pump when the hot-catch-pot level was allowed to become too low.

Gas flows - These were steady. Hydrogen consumption was steady. There was one 15-minute period when the gas flow was reduced by about 40% because of check-valve failure on the east recycle-gas compressor.

Temperatures - These were steady during the run. The RO pump-head was kept at 400-410°C during the run.

Pumps and Compressors - There was one failure of the east recycle-gas compressor. The feed-pump and RO-pump operations were good.

Cause of shutdown

This run was shut down because of an electrical failure. The electrician was doing work for the Technical Services Division (installation of a timer on an air compressor) when the fuse "blew" on the 110-V transformer which operates all of the switching relays. This shut off the recycle-gas compressor for 5 minutes. After the fuse was replaced the air compressor would not run and, while wires were being replaced to make it operate, the transformer fuse "blew" again. After a total of 10 minutes with liquid but no recycle gas in the reactor, shutdown procedures were carried out.

Inspection after Completion of Run

Reactor - The reactor walls had a light dusting of powdery material which was removed by 3 or 4 brushings. The top cap was clean. The bottom cap was 3/4 full, with pieces of hard black material on top of a hard, porous base in the cone of the cap.

Hot receiver (hot catch pot) - The hot receiver had a light dusting of powdery material. The top cap was clean. The bottom cone had a 1-mm-thick coating of hard black material.

Light ends receiver - Clean.

Gas lines - Clean.

Traps before compressor - Clean.

Preheaters - Clean.

Down pipe in HCP - Clean.

Discussion

The operation of the plant was smooth at these conditions. The cause of the shutdown was electrical failure and the operation time was too short, hence nothing should be said about operational difficulties.

Conclusion

The run would have continued for a longer period. Comparison with later runs will determine whether the material in the bottom reactor-cap was caused by the period without hydrogen or by the run conditions.

Recommendations

1. Repeat the run at similar conditions.
2. Reduce the RO-pump section line-temperature.

March 26, 1976 RUN NO. R-2-2-2 10 hours

Conditions

Same as for run R-2-2-1.

Purpose

Same as for run R-2-2-1.

Changes since Previous Run

The temperature of the RO-pump section was reduced to 250-270°C. This was accomplished by removing the insulation on the HCP-liquid discharge line to the RO-pump-section line and the Ruska pumps.

Preparation for Run

1. General clean-up.
2. Removed the insulation between the HCP-liquid discharge line to the RO and the Ruska pumps.

Start-up

Same as for run R-2-2-1.

Operations

Operation was normal for about 8 hours. Then for about two hours, until the shutdown, the pressure drop was swinging between 60 and 140 psi.

Cause of Shutdown

A line plug which caused a 1200-psi pressure-drop developed after about 10 hours of operation at 460°C in the section between the reactor and the compressor discharge, since before and after the reactor was dumped there was a high pressure-drop with the gas on either recycle or by-pass. With the recycle gas on by-pass, there is no flow through the reactor, the HCP or the light-ends system, and the gas simply circulates through a portion of the line from the oil scrubber and a portion of the line which leads to the reactor, shown as a dashed line in Figure 1. Since the pressure-drop was high in this line, plugging occurred here.

Inspection after Completion of Run

The next run was started immediately after the shutdown; therefore no inspection was made after this run.

Discussion

Before the shutdown the overall pressure-drop was fluctuating. This may have been caused by heavy-oil carry-over to the light-ends receiver, or by bitumen feed backward-flow into the gas-recycle line. If heavy oil is carried over from the hot separator (HCP) because of high level or foaming, it will cause plugging since it becomes very viscous at the low temperature in the light-ends system. Feed could flow backwards into the gas-recycle line because of pressure fluctuations in the system.

Conclusion

The gas-recycle or by-pass line was plugged by the heavy-oil carry-over but it was not plugged solidly and it cleared itself.

Recommendations

This run should be repeated immediately without any clean-up or inspection. A check-valve should be installed so that bitumen feed cannot flow backwards into the gas-recycle line.

March 27, 1976 RUN NO. R-2-2-3 7 hours

Conditions

Same as for run R-2-2-2, except for the RO-pump stroke, which was set at 1.5 inches.

Purpose

Repeat run R-2-2-2.

Changes since Previous Run

None.

Preparation for Run

None.

Start-up

The run was started immediately after the shutdown of run R-2-2-2.

Operation

Operation was normal until plugging. There was no advance warning.

Cause of Shutdown

A line plug developed seven hours after the start of feeding and this shut down the run.

Inspection after Completion of Run

The reactor, the HCP, the transfer lines and the preheaters were clean. There was a plug of black material in the cooler situated before the light-ends receiver. It was apparent that this was the cause of the shutdown as there were no other plugged lines.

Discussion

There was no warning of the plugging, which forced a shutdown in a few minutes. From the inspection it is clear that the cause of the shutdown was the plug in the LE cooler. This could also have caused the apparent symptom of plugging in the reactor, the HCP and the transfer lines, since the HCP pressure-gauge is situated down-stream from the cooler. The reason for the plug could have been a partially plugged line on start-up of run R-2-2-3.

Conclusion

For all previous recycle runs it is apparent that heavy-oil carry-over into the light-ends receiver was the rule, rather than the exception. The five-day run R-2-1-1 was shut down because of heavy-oil carry-over. The 20-day run R-2-1-2 was successful, but clean-up revealed some heavy-oil carry-over. Finally, run R-2-2-2 was shut down possibly because of carry-over, and run R-2-2-3 was shut down definitely because of carry-over.

Recommendations

Perform clean-up and repeat the run again. Increase the size of the four holes in the HCP dip-tube nozzle to 1/8 in. (3.18 mm) from 1/16 in. (1.6 mm) to reduce the jetting effect and foam formation.

March 30 - April 10, 1976 RUN NO. R-2-2-4 283 hours

Conditions

Same as for run R-2-2-1.

RO recycle rate = 12,700 g/h

Purpose

Same as for run R-2-2-1.

Changes since Previous Run

None.

Preparation for Run

1. Complete clean-up.
2. The HCP dip-tube-nozzle holes were changed from 1.6 mm (4 holes) to 3.18 mm size.

Start-up

Liquid feed was started when the temperature of the reactor was 350°C. The RO pump was started to recirculate the HE. In order to control the HCP level, the feed rate was adjusted. The temperature of the reactor was increased rapidly to 430°C. After 430°C, the temperature of the reactor was increased slowly (5° rise in temperature every hour) to 460°C. At this temperature the feed rate was adjusted to 4500 g/h and the Ruska pump was started and adjusted to maintain the HCP level.

Operation

Pressure drop - The system pressure-drop increased steadily to 250 psi in about 12 days, but the variation in the system pressure-drop was not too great when compared to run R-2-1-2 where the HCP level was controlled by adjusting the feed rate.

Feed and product flows - Feed flow varied between 4300 g/h and 4800 g/h with an average value of 4556 g/h. The daily-average value of feed was nearly constant. The daily averages of LE and RO were also nearly constant throughout the run.

Gas flows - Throughout the run there were problems with the gas-recycle compressors. The gas flow-rate would slow down or become erratic because of check-valve failure. There were three problems:

1. Springs in the check valve would become inoperative because of corrosion.
2. There was a deposit of black material in the valves, which was probably dessicant from the trap situated upstream from the compressor.
3. The third problem was with the lubricating-oil supply to the recycle-gas compressors. The spare recycle-gas compressor used much more oil than the new compressor and therefore the oil-supply vessel did not have adequate capacity. It was filled while the plant was on-stream. The gases dissolved in the oil came out of solution and may have caused air-lock problems in the oil-feed line.

Temperature - All the temperatures were steady.

Pumps and controls - Apart from the recycle-gas compressor, the oil-scrubber pumps were leaking badly. The left piston was badly scored and was replaced.

Cause of Shutdown

The reason for shutdown was oil pumped into the gas lines from the hydrogen-supply vessel (operator's error).

Inspection after Completion of Run

Reactor - The top reactor-cap had a small amount of bubbly oil. The reactor bottom-inlet cap was clean. The reactor wall and the thermocouples were coated with black powder.

HCP - The crossover line contained a heavy deposit. The HCP vessel had a very thin layer (about 0.08-mm thick) in the lower 1-meter portion. The down-tube was almost plugged but the outlet cap had very little oil on it.

Light ends receiver - This vessel had small amounts of black slurry (1/4 pint) at the bottom.

Traps before the compressors - The traps had a purplish substance in the dessicant. It was not much different from in the other runs.

Preheaters - Preheater number 1 was clean, but numbers 2 and 3 (the hottest preheaters) had some solid on the walls.

Down-pipe in the HCP - This had a thin layer of black deposit on the outside of the upper half and it was thicker at the bottom. The holes were fully open.

Other problems - The left piston of the oil-scrubber pumps was badly scored. Small amounts of oil were present in the gas line from the scrubber to the gas pumps.

Discussion

The system pressure-drop increased to 250 psi in about 12 days, but the fluctuations in the pressure drop were not too large. The feed, LE and RO productions were nearly constant. The fluctuations in ΔP, feed rate, LE and RO production rate were much less than for run R-2-1-2 in which the HCP level was controlled by adjusting the feed rate. After shutdown the deposits in the system were also less compared to those for run R-2-1-2.

Conclusion

Although the system pressure-drop increased to 250 psi, the run could have been operated for a longer period of time. Control of the HCP level by adjusting the RO withdrawal-rate seems to be better than by adjusting the feed rate.

Recommendations

1. Use stainless-steel springs in the gas-recycle compressors.
2. Install stainless-steel screen supports for the dessicant instead of Fiberglas supports in the trap on the suction side of the gas-recycle compressors.
3. Install a by-pass on the traps.
4. Install oil reservoirs and a pump so that lubricating oil can be pumped into the recycle-gas compressor while in operation.
5. With these modifications, runs should be tried at similar conditions, at different LHSV's and at different temperatures.

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