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ZINC CHLORIDE CATALYZED FLASH HYDROPYROLYSIS OF

+ 525 °C PITCH FROM COLD LAKE BITUMEN

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$ZnCl_2$ CATALYZED FLASH HYDROPYROLYSIS OF +525 $^{\circ}C$ PITCH FROM COLD LAKE BITUMEN

by

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ABSTRACT

Flash hydropyrolysis experiments have been performed on the vacuum bottoms fraction of Cold Lake bitumen, using zinc chloride as a catalyst. It was found that high conversions could be obtained at hydrogen pressures which are much lower than those normally used in catalytically hydrocracking residual oils.

RESUME

Des essais ont été effectués utilizant le résidu de distillation du bitume de Cold Lake en présence du chlorure de zinc comme catalyzeur. On a trouvé que des conversions hautes peuvent être réalisées à des pressions d'hydrogène beaucoup plus basses que celles qui sont necessaire pour le hydrocraquage des braies.

For many years delayed coking and fluid coking have been the only industrial processes used to upgrade the bitumen derived from Canada's oil sand resources. The hydrocracking process units that are currently being constructed are considered to be the next generation of upgraders. In spite of the success of these commercial processes, alternatives that might have potential advantages merit consideration. Experiments with one such alternative, catalytic flash hydropyrolysis, were performed in order to examine its characteristics. Either pitch from the original bitumen or the unreacted pitch by-product from a bitumen hydrocracking process can be used as the feedstock for a catalytic flash hydropyrolysis unit.

During flash hydropyrolysis the feedstock is heated rapidly to the reaction temperature in the presence of hydrogen, is kept at reaction conditions for a short period and, is cooled quickly before extensive secondary reactions can occur. Considerable work describing flash pyrolysis (Scott et al., 1986) and flash hydropyrolysis of coals (Hiteshue et al, 1957; Stangeby and Sears, 1981a) has been reported. Unfortunately, flash hydropyrolysis experiments with bitumen and heavy oils have been less extensive.

Bunger et al. (1981) performed one non-catalytic flash hydropyrolysis experiment on each of three different feedstocks derived from Utah Oil Sands. Extremely large gas velocities, 700,000 scf/Bbl (124.7 m^3 H_2/m^3 feed), were used. Subsequently, Bunger (1985) reported a single non-catalytic flash hydropyrolysis experiment on Athabasca bitumen. Stangeby and Sears (1981b) used oil sand samples (bitumen plus the original sand) in their flash hydropyrolysis experiments. Walsh and Chen (1983) measured the effects of several experimental variables: temperature, hydrogen pressure, vapour residence time and feedstock hydrogen to carbon ratio while studying the non-catalytic hydropyrolysis of a heavy petroleum oil. The lowest char yield they obtained was 18 wt %.

Shabtai et al. (1979) described a general reaction mechanism for flash hydropyrolysis. They suggested that the initiation step is the same as in thermal cracking. Free radicals can be formed from homolytic cleavage of a C-C bond.

$$R_1\text{-}R_2 \quad \text{---->} \quad R_1{}^{\bullet} + R_2{}^{\bullet} \hspace{4cm} (1)$$

The explanation for the reaction rates in hydropyrolysis being greater than those in thermal cracking involves the formation of hydrogen atoms, $H^\bullet$. Atomic hydrogen is produced when some of the free radicals formed by Equation (1) are stabilized, by interacting with molecular hydrogen (H_2).

$$R^\bullet + H_2 \dashrightarrow RH + H^\bullet \tag{2}$$

Comparatively few hydrogen atoms will be formed directly from molecular hydrogen via Equation 3.

$$H_2 \dashrightarrow 2 H^\bullet \tag{3}$$

The reason Equation 3 is not favourable becomes apparent by considering bond energies. Typical bond energies are: 347 kJ/mol for a C-C bond (Equation 1), 435 kJ/mol for a H-H bond (Equation 3), 414 kJ/mol for a C-H bond and (435 - 414) = 11 kJ/mol for Equation 2. It is obvious that most hydrogen atoms will be formed by Equation 2 (11 kJ/mol) rather than Equation 3 (435 kJ/mol).

The difference between Equation 1 (347 kJ/mol) and Equation 3 (435 kJ/mol) is also enormous when considered in terms of free radical concentrations. Both Equations 1 and 3 can be written in the form

$$A \dashrightarrow 2 B^\bullet \tag{4}$$
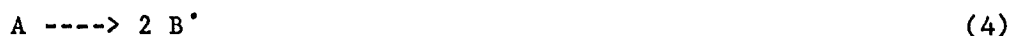
The equilibrium constant for this reaction can be written as

$$K = (y_{B^\bullet})^2 / y_A = \exp(-E_B/RT) \tag{5}$$

where E_B is the sum of the energies for bond forming and bond breaking in a particular reaction. When Equations 1 and 3 are compared in this manner, it is apparent that $y_{B^\bullet}$ is several orders of magnitude larger when $B = R^\bullet$ than when $B = H^\bullet$. This is caused by E_B being in an exponent term.

The hydrogen atoms propagate the radical chain reaction by abstracting

hydrogen from other hydrocarbon molecules to form secondary radicals plus molecular hydrogen. This can be shown as Equation (2) written in the reverse direction.

$$H^{\cdot} + RH \longrightarrow R^{\cdot} + H_2 \tag{6}$$

If suitable processing conditions are chosen a steady state involving reactions 2 and 6 will be established. Subsequently, secondary radicals will undergo beta cleavage to form an olefin and another radical.

$$R_1-CH_2-CH_2-CH_2-R_2^{\cdot} \longrightarrow R_1-CH=CH_2 + CH_3-R_2^{\cdot} \tag{7}$$
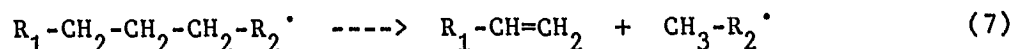
It is the combination of reactions 6 and 7 which causes the hydropyrolysis reaction rates to be greater than those of thermal cracking. Nevertheless, the combination of all the above reactions is required for the chain reaction.

The desirable products from flash hydropyrolysis are distillate hydrocarbon oils. Unfortunately some solid coke or char is often produced as an unwanted by-product. This occurs when free radicals polymerize into higher molecular weight species. Coke formation can be inhibited by enhancing the radical stabilization reaction, Equation 2. Equation 7 needs to proceed without coke being formed from extensive polymerization between radicals. This implies that most of the radicals must either undergo beta cleavage via Equation 7 or be stabilized via Equation 2 before they encounter other hydrocarbon radicals and form coke.

The purpose of the work described here is to extend the previous non-catalytic work (Bunger, 1981; Bunger, 1985; Walsh and Chen, 1983) on bitumen and heavy oils to reaction systems which include a catalyst. Flash hydropyrolysis experiments were performed on +525 °C pitch (vacuum bottoms from Cold Lake bitumen which boils at temperatures higher than 525 °C) using zinc chloride as the catalyst.

Experimental

A schematic diagram of the flash hydropyrolysis reaction system is shown in Figure 1. In a typical experiment, nominally 50 mg of pitch was mixed with catalyst to give the desired catalyst to pitch ratio, and then placed in an aluminum boat. The aluminum boat was placed in a stainless steel tubular reactor 0.375 m long and 4.75 mm

inside diameter. The reaction tube was heated rapidly by passing a large current through it. The current was controlled by solenoids activated by timers. The primary heating system was controlled by the first timer and produced heating rates from 100 to 650 ^{o}C/s. After the preset temperature was attained, the secondary heating current, which was controlled by a second timer, maintained the reactor at constant temperature for the desired time.

Two type K thermocouples were located at different positions on the reactor exterior. The millivolt signals from the thermocouples were connected to a model 2213A Tektronix dual channel oscilloscope. They were displayed as a function of time, and were photographed using a Tektronix C-5C oscilloscope camera. Both temperature profiles and heating rates were determined from the photographs. An example is shown in Figure 2. The thermocouples were calibrated by determining the millivolt signals at the melting points of zinc, aluminum and copper metals.

Heat transfer effects were examined in two ways. The rate of heat transfer through the reaction tube was examined by comparing the temperature of a thermocouple inside the reactor with one outside. The one inside the reactor had its junction dipped into the pitch contained in the aluminum boat. The two temperatures were found to be within 10 ^{o}C. The sample size (pitch plus catalyst) was varied from 50 to 200 mg to determine its effect on temperature profile and heating rate. Similar results were obtained in all cases.

The properties of the + 525 ^{o}C pitch are listed in Table 1. Reagent grade zinc chloride was obtained from Caledon Laboratories. High purity hydrogen gas was obtained from the Matheson Company.

During an experiment hydrogen flowed continuously through the reactor and swept the reaction products into the sample collection system. Condensable products were collected in a coiled steel cold trap at - 50 ^{o}C. The gaseous products flowed through the cold trap into an evacuated cylinder. The solid residue (unreacted pitch plus char) remained in the aluminum boat.

After the experiment the gases were analyzed using a gas chromatograph and the condensed materials in the cold trap were divided into two fractions, a light distillate and a heavy distillate. After the hydrogen flow was diverted, to an

adsorption trap filled with 6-14 mesh Fisher activated carbon, the cold trap was heated to 100 $^\circ$C to remove the light distillate. In the next step, carbon disulphide was used to extract the light distillate components which had been adsorbed on the activated carbon. The resulting solution was analyzed by gas chromatography. This technique was verified by placing known quantities of hydrocarbon mixtures on the activated charcoal. The recoveries exceeded 90 %. The heavy distillate products remaining in the cold trap were extracted with carbon disulphide and acetone. These solvents were evaporated and the extracted hydrocarbons weighed.

Several analyses were performed on the heavy distillate and on the solid residue. Carbon, hydrogen and nitrogen analyses of the dried heavy distillate were performed using a model 240B Perkin Elmer elemental analyzer. A known weight of liquid sample was combusted at 1000 $^\circ$C in the presence of oxygen. The product gases flowed sequentially through both an oxidation furnace and a reduction furnace. The resulting CO_2, H_2O and N_2 were analyzed chromatographically and the % C in the sample calculated. NBS certified materials acetanilide, phenacetin, cyclohexanone and 2,4 dinitrophenyl hydrazine were used to calibrate the instrument. Oxygen determinations were made with a modified configuration of the same instrument. Sulphur in the original pitch sample was determined by combusting the pitch in a closed flask filled with oxygen. All sulphur was converted to sulphate by hydrogen peroxide and the sulphate titrated with barium perchlorate. Simulated distillations of the heavy liquids were performed in accordance with ASTM Method D2887.

The solid residue left in the aluminum boat at the end of an experiment was combusted at 800 $^\circ$C with oxygen. The carbon dioxide produced was analyzed by gas chromatrography to determine the % C.

Results and Discussion

Figure 3 shows the effect of temperature on the product distribution. At low temperatures the conversion is low and the solid residue is probably composed primarily of unreacted pitch rather than char. Char is considered to be a solid reaction product containing aromatic and aliphatic carbon atoms in condensed rings plus alkyl groups. It can only react further by dealkylation to form gases and by dehydrogenation to form additional aromatic carbon atoms, resulting in a char having

more highly condensed ring structures. Unreacted pitch is that material which can react further (via primary reactions) to produce distillate oil as well as char and gases. As the temperature increases the heavy distillate liquid yield goes through a maximum, as shown in Figure 3. The decrease in heavy distillate at the highest temperature can be explained in terms of secondary reactions. Secondary reactions are those in which the desirable distillate products react further to form gases and char. This is illustrated by the simplified reaction network shown in Figure 4. The increase in gas and light distillate formation shown in Figure 3 could result from primary reactions at low temperature and secondary reactions at high temperature. For constant residence time and increasing temperature (Figure 3) an increase in secondary reactions is expected. Therefore the maximum in heavy distillate yield can be explained by a combination of heavy distillate formation by pitch conversion (significant at low temperatures) and heavy distillate consumption by secondary reactions (significant at high temperatures).

Table 2 compares results obtained with a $ZnCl_2$ catalyst with those obtained without a catalyst. In both cases the carbon recovered in the products exceeded 93 % of that in the feedstock. It is apparent that the catalyst increased the total distillate yield and decreased the yield of solid residue. This is consistent with earlier work in which Nakatsuji et al (1977) and Zielke et al. (1966b) indicated that the $ZnCl_2$ catalyst enhances both cracking and hydrogenation reactions. Enhanced cracking reactions (Equations 1 and 7) would increase the distillate oil yield whereas enhanced hydrogenation reactions (Equation 3) would decrease the solid residue yield.

Figure 5 shows the effect of the catalyst to pitch ratio on the product distribution. The total conversion decreases slightly as the catalyst to pitch ratio increases from 1 to 2. Earlier work by Zielke et al (1966a) had shown that conversion increased substantially as the catalyst to feedstock ratio increased from 0 to 1. The major effect at the highest catalyst to pitch ratio in Figure 5 is the decreased yield of heavy distillate and the increased yield of all other products. This suggests that secondary reactions may result when there is a larger amount of catalyst which can contact a fixed amount of heavy distillate.

Several studies have described the interaction of the catalyst with the feedstock. In hydrocracking studies (not flash hydropyrolysis studies) the metal

chloride catalyst was found to form a complex with the carbonaceous feedstock (Zielke et al, 1966b; Morita and Hirosawa, 1975). Studies with both $ZnCl_2$ (Zielke et al. 1981; and Nakatsuji. et al 1977), and $SnCl_4$ (Kriz et al. 1979) have shown that the metals in the catalyst-carbon complex are present as sulphides, not chlorides. Zielke et al. (1980) found that there was a decrease in the carbon content of the spent complex (reactor residue) as the conversion of the feedstock increased.

The above observations are consistent with the catalyst providing sites which interact with the carbon material while reaction occurs. As more catalyst becomes available (greater catalyst/pitch ratio in Figure 5) additional sites are provided to interact with the carbonaceous material. If the carbonaceous material remains in the catalyst-complex too long, there will be a greater possibility of secondary reactions. This might be the cause of the decreased yield of heavy distillate shown at the greatest catalyst/pitch ratio in Figure 5.

Kershaw, Barrass and Gray (1980) studied flash hydropyrolysis of coal with $SnCl_4$. They found that both the molecular weight and the viscosity of the product oil decreased with increasing catalyst concentration in the reactor. The infra-red spectra of the product oil showed that the intensity of the peak at 3380 cm^{-1} (hydrogen bonded OH) decreased as the catalyst concentration increased. Increasing the quantity of catalyst may have provided additional reaction sites in the catalyst-carbon complex for conversion of these OH groups. The observed decrease in viscosity of the product oil would be expected if hydrogen bonding in the oil decreased, as a result of the removal of hydrogen bonded OH groups.

Slightly different final temperatures were used in some of the experiments shown in Figure 5. If the final temperature is too high, it can adversely affect the heavy distillate yield. In this respect the data in Figure 5 are completely consistent with those in Figure 3.

Figure 6 shows the influence of heating rate on the product distribution. The conversion goes through a maximum and the solid residue yield goes through a minimum as the heating rate is increased. When the heating rate is slow, the temperature will increase slowly and high molecular weight free radicals, in the dense phase (solid plus liquid), will be at reaction conditions for a comparatively long time before they are cracked and volatilize. This will provide an opportunity for free radicals to

polymerize, which in turn would increase the amount of char formed. The greater amount of residue shown in Figure 6 at the slowest heating rate, is consistent with the above explanation. At somewhat faster heating rates, the temperature will increase more quickly and the higher molecular weight free radicals will pyrolyze at the higher temperatures before they polymerize. The pyrolysis products will volatilize from the dense phase to the vapour phase and leave the reactor. There would be less opportunity for free radical polymerization in the dense phase since many free radicals would have been swept out of the reactor after they had volatilized. At the fastest heating rate, the temperature may have increased faster than the rate at which the pyrolysis products could volatilize and leave the reactor. Consequently, secondary reactions could have occurred to the pyrolysis products. Secondary reactions can explain the results at the fastest heating rate in Figure 6 as the heavy distillate oil yield decreases and the residue, gas and light distillate yields increase.

Figure 7 shows that the vapour phase residence time of the products in the reactor vapour phase has only a small influence on the product distribution. The initial increase in conversion with increasing residence time may be attributed to a slightly greater concentration of hydrogen atoms in the vapour phase, as shown in Equations 2 and 3. The subsequent decrease in conversion and in distillate yield at the greatest residence time can be attributed to secondary reactions involving hydrocarbon free radicals formed from hydrogen atoms.

Figure 8 shows that the hydrogen pressure had almost no effect on the product distribution. Only at the highest pressure was there a slight decrease in conversion. A small but continuous decrease in distillate yield occurred with increasing hydrogen pressure. This may have been caused by the higher concentration of hydrogen which saturated the hydrocarbon radicals (Equation 2) before beta splitting occurred.

In spite of the small quantities of product samples, some quantatative analyses were performed. The gas analyses indicated that the principal products were C_1 to C_5 hydrocarbons. The concentrations of some gaseous products are shown in Figure 9, as a function of heating rate. At the higher severities, the concentrations of methane and ethane increase dramatically. Perhaps this represents increased dealkylation. Concentrations of propane, ethene and propene were less sensitive to severity. The light distillates were analyzed by gas chromatography.

The principal components were benzene, toluene and xylene, (BTX). The boiling point distribution curve for one of the heavy distillate products is shown in Figure 10. It is readily apparent that the molecular components are fairly uniformly distributed with boiling point, throughout the heavy distillate.

Conclusions

Conceptually, , it would appear that short pitch residence times are necessary to avoid polymerization and that short product residence times are necessary to avoid secondary reactions. The flash hydropyrolysis process attempts to satisfy these criteria. In order to have high conversions at short residence times, very fast heating rates are essential. However, the work reported here shows that if the heating rates are too fast, secondary reactions will occur before volatilization. Similarly, if the final reaction temperature is too high, secondary reactions will occur before the products leave the reactor. While the catalyst enhances both cracking and hydrogenation reactions, excessive amounts will also enhance secondary reactions.

The fact that the hydrogen pressure seems to have only a minor influence on the product distribution is important. It suggests that a flash hydropyrolysis process might produce reasonably high conversions at pressures as low as 1.8 MPa (250 psig). This pressure is almost an order of magnitude lower than those being used in commercial processes to hydrocrack residual oils. If lower pressures could be used, there might be a significant decrease in the investment capital required to construct an upgrading plant.

Corrosion from chloride catalysts is a genuine concern. However, if the capital cost saving from decreased pressure was sufficient, special corrosion resistant materials might be justified in the low temperature portion of the plant where condensation of moisture could occur.

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List of Captions

Figure 1 Schematic Diagram of Reaction System.

Figure 2 Heating Rate Measurement - Temperature (C) Versus Time (s).

Figure 3 The Effect of Temperature on Product Distribution. (Heating rate = 237 ^{o}C/s; Hydrogen pressure = 7 MPa; Residence time = 3.95 s; Catalyst/pitch ratio = 0.93).

Figure 4 Reaction Network. Reactions consist of pyrolysis-polymerization, hydrogenation-dehydrogenation, and dealkylation.

Figure 5 The Effect of Catalyst/Pitch Ratio on Product Distribution. (Heating rate = 237 ^{o}C/s; Hydrogen pressure = 7 MPa; Nominal final temperature = 550 ^{o}C; Residence time = 3.95 s). Open data points represent final temperatures of 510 - 520 ^{o}C. Solid data points represent final temperatures of 580 - 590 ^{o}C.

Figure 6 The Effect of Heating Rate on Product Distribution. (Hydrogen pressure = 7 MPa; Nominal final temperature = 550 ^{o}C; Residence time = 3.95 s; Catalyst/pitch ratio = 0.93)

Figure 7 The Effect of Residence Time in the Vapour Phase on Product Distribution. (Heating rate = 237 ^{o}C/s; Hydrogen pressure = 7 MPa; Nominal final temperature = 550 ^{o}C; Catalyst/pitch ratio = 0.93).

Figure 8 The Effect of Hydrogen Pressure on Product Distribution. (Heating rate = 237 ^{o}C/s; Nominal final temperature = 550 ^{o}C; Residence time = 3.95 s; Catalyst/pitch ratio = 0.93).

Figure 9 The Effect of Heating Rate on the Distribution of Gaseous Products. (Hydrogen pressure = 7 MPa; Nominal final temperature = 550 ^{o}C; Residence time = 3.95 s; Catalyst/pitch ratio = 0.93).

Figure 10 Boiling Point Distribution Curve for a typical Heavy Distillate (Heating rate = 237 C/s; Hydrogen pressure = 500 psig; Nominal final temperature = 550 C; Residence time = 4 s; Catalyst/pitch mass ratio = 0.93)

TABLE 1

PROPERTIES OF + 525 $^{\circ}$C PITCH (VACUUM BOTTOMS)

FROM COLD LAKE BITUMEN

	Wt %
Carbon	82.5
Hydrogen	10.4
Sulphur	5.5
Nitrogen	1.1
Oxygen (by expt)	1.2
Ash	0.2
+ 525 $^{\circ}$C Pitch	80.0
Pentane insolubles	49.2
Benzene insolubles	0.0
Toluene insolubles	0.1
Average Molecular Weight	
Vapour pressure osmometry	841
Freezing point depression	817

TABLE 2

COMPARISON BETWEEN

NON-CATALYTIC AND CATALYTIC EXPERIMENTS

PROCESSING CONDITIONS

Heating Rate ^{o}C/s	237	237
Hydrogen Pressure MPa	7	7
Final Temperature ^{o}C	582	523
Residence Time s	3.95	3.95

CATALYST/PITCH RATIO	0.0	0.927

CARBON AS % OF ORIGINAL PITCH

C_1	8.5	4.0
C_2's	11.5	4.2
C_3's	5.7	6.2
C_4's	0.8	1.5
C_5's	1.1	0.9
Light Distillate	8.1	2.6
Heavy Distillate	16.3	67.1
Residue	41.6	7.3
TOTAL	93.6	93.8
Losses	6.3	6.2

He (purge)

H_2

PI   PI

PRV

OSC

TI   TI

STAINLESS STEEL
REACTOR

SAMPLE

VAPOUR
RESIDENCE
ZONE

VENT

GAS
Coll-
ector

FI

GC

Carbon
Trap

COLD
TRAP