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CANMET HYDROCRACKING AND COPROCESSING FOR SYNTHETIC FUELS PRODUCTION

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ABSTRACT

The CANMET hydrocracking and coprocessing technologies have been reviewed in terms of feedstocks and product characteristics. An overview of both technologies are presented. The effect of process severities on compound-type distributions are also discussed.

The main objective of this report is to provide information on the compound-types present in synthetic fuels that require further treatment in order to meet product specifications and environmental requirements.

PROCÉDÉS D'HYDROCRAQUAGE ET DE COTRAITEMENT CANMET POUR LA PRODUCTION DE COMBUSTIBLES SYNTHÉTIQUES

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RÉSUMÉ

Les technologies d'hydrocraquage et de cotraitement CANMET ont été étudiées au niveau des charges d'alimentation et des caractéristiques du produit. Un aperçu des deux procédés est présenté dans le présent rapport. On y examinera, de plus, l'effet de la sévérité du procédé sur les distributions d'hydrocarbure.

L'objectif principal de ce rapport est de fournir des renseignements sur les hydrocarbures présents dans les combustibles synthétiques qui ont besoin davantage de traitement afin de satisfaire aux spécifications du produit et aux exigences environnementales.

INTRODUCTION

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In the past, upgrading of bitumens, heavy oils and petroleum residues required the use of pyrolytic (coking) processes. These processes were wasteful because they produced large quantities of coke which had to be stockpiled. In response, CANMET, Energy, Mines and Resources Canada has developed an alternative method known as CANMET hydrocracking coking, sought by CANMET, whereby more than 90% of the feedstock is converted to synthetic crude. In hydrocracking, the H/C ratios of various low-grade feedstocks are increased at high hydrogen pressure and temperature in the presence of a coal-based disposable additive. The CANMET hydrocracking process has been shown to be economically superior to other upgrading processes and is currently in the demonstration stage, 5000 bb/d capacity at the Petro-Canada refinery in Montreal.

CANMET coprocessing replaces a portion of heavy oil/bitumen or residuum by less expensive coal while the operating conditions and additive remain virtually the same. CANMET is a pioneer in coprocessing and for the last five years has established a large database using Alberta coals and heavy oils/ bitumens and other conventional vacuum residues. CANMET coprocessing involves simultaneous upgrading of heavy oil and coal in a once-through operation. The recycle solvent required in the coal liquefaction process is eliminated in the CANMET process. A comparison shows that CANMET coprocessing is superior to liquefaction and comparable to hydrocracking in terms of product yields and qualities.

This paper discusses the general characteristics of some feedstocks and their hydrocracked and coprocessed products. Although a large database is available, the main focus is on the coprocessing of Forestburg subbituminous coal and Cold Lake vacuum bottoms. Where data on Cold Lake vacuum bottoms available, the results from different feedstocks are presented. Emphasis is primarily on the content of aromatic components and the heteroatoms in both feedstocks and products. The effect of process severity on aromatic compound conversions and heteroatom removal is also discussed.

CANMET HYDROCRACKING

Process Description

A simplified schematic of the CANMET hydrocracking process is shown in Fig. 1. The additive (pulverized coal impregnated with iron sulphate or other metallic salts) is prepared separately and slurried with the feedstock. Slurry feed combined with recycled hydrogen are heated and fed into the bottom of an up flow reactor. The products leave the top of the reactor and are separated into heavy oil, light oil, and gas fraction. The gas is purified by removing hydrocarbons and hydrogen sulphide, then recycled to the reactor with make-up hydrogen. The oil products are fractionated into suitable fractions for further hydrotreating.

Feedstocks

During the last seven years more than 50 000 h of operation have been achieved in two pilot plants (1 bbl/d) at CANMET's Energy Research Laboratories. The gross properties of some of the feedstocks processed to date in the CANMET hydrocracking mode are shown in Table 1. All feedstocks, except Boscan and Lloydminster contain high concentration of impurities such as sulphur, nitrogen and metals which would be problematic for catalytic upgrading processes. The distillated yields from the coking processes using the feedstocks listed in Table 1 would probably be low since approximately 20 wt % coke is produced as byproduct which cannot be used as fuel because of its low H/C ratio.

Chemistry of Hydrocracking

Hydrocracking of heavy oils/bitumen or residuum involves the breakdown of large molecules at relatively high temperatures and capping of the generated radicals by hydrogen in the presence of a catalyst. The chemical structures of heavy oils/bitumens are very complicated. Detailed characterization of these materials requires sophisticated instrumentation which is time consuming. Suzuki et al. carried out a detailed study on the molecular structure of Athabasca, Cold Lake and Orinoco bitumens using ¹H and ¹³C n.m.r.(1). These bitumens were separated into distillates and residues by vacuum distillation. Then each distillate and residue was fractionated into saturates, aromatics, resins and asphaltenes. The average structure of each

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of these fractions is shown in Fig. 2. and 3. Comparison of the structural parameters obtained by 13 C n.m.r. analysis indicated that Cold Lake and Athabasca bitumens are very similar. However, it is not always possible to carry out such an extensive and detailed characterization on either feedstocks or products. Most laboratories evaluate a process performance by classifying materials according to their solubility in different solvents. Oils are defined as pentane solubles, asphaltenes are pentane insolubles but toluene solubles and preasphaltenes are toluene insolubles but tetrahydrofuran (THF) solubles.

Hydrocarbon-type analysis is another technique widely used in the petroleum industry for chemical characterization of feedstocks and products. Most laboratories use high performance liquid chromatography for class separation. A reliable method for hydrocarbon-type separation was developed in the USBM-API Project 60 (2) and has been modified at CANMET (3). Using the separation technique and according to the schematic diagram shown in Fig. 4 the Alberta basin bitumens and crude oils were characterized (4).

Table 2 lists the sample feedstocks in order to their burial depth. The total PAH content in the deasphaltened residue boiling above 350°C decreased from 23.2 wt % to 7.6 wt % whereas the total saturates increased from 19.4 wt % to 56.0 wt %. Results of further separation and characterization of PAH ring classes from these bitumens and oils are shown in Table 3. The 3-ring components such as phenanthrenes and anthracenes increase gradually with increasing burial depth. There is also a small increase in the 4-ring aromatics such as pyrenes and chrysenes. The increase in 3 and 4 aromatic rings as a function of burial depth appears to be at the expense of PAH containing more that 4 fused rings per molecule such as benzo [a] pyrenes, perylenes and dibenzo [a,h] anthracenes which decrease from 25 to 15 wt %. When Athabasca bitumen was hydrocracked at relatively high severity, the total PAH content was reduced from 23.2 to 16.2 wt % and the 3- and 4-ring structures increased whereas molecules containing more that 4 fused ring decreased.

Figure 5 shows how process severity affects compound-type distribution in hydrocracking of Cold Lake vacuum bottoms. As the total saturates and to some extent mono and di-aromatics increased as a function of severity, poly-aromatics and asphaltenes decreased.

Table 4 lists the components identified in the polynuclear aromatic hydrocarbon fraction of Cold Lake hydrocracked product boiling above 350°C. In total, 97 polynuclear aromatic hydrocarbons were identified but only those having concentrations higher than 50 ppm are listed in Table 4 (5). It was also indicated that as the weight per cent of distillation residue above 350°C increased the PNA content also increased. For example, Cold Lake with a 350°C distillation residue of 98.1 wt % contains 19.2 wt % PNA whereas Medicine River with a 350°C distillation of residue of 38.3 wt % contains 7.2 wt % PNA.

One of the advantages of the hydrocracking process over the coking process is the removal of heteroatoms such as sulphur and nitrogen. Further removal of different sulphur and nitrogen compounds in the hydrocracked liquid product by hydrotreating depends on the type and general structure of these compounds. A knowledge of the total heteroatom content in products is important, however, the chemical group composition is also very important as some sulphur structures are more harmfull than others for certain applications. Table 5 shows the sulphur-type distribution in the naphtha product obtained in hydrocracking of Athabasca bitumen (6). The percentage sulphur content decreased as the severity of hydrocracking increased. The main sulphur types in the naphtha product are residual sulphur, aliphatic sulphides and aromatic sulphides.

The nitrogen content of the nitrogenous compound-type concentrates in Athabasca bitumen and hydrocracked products using anion and cation exchange columns is shown in Table 6. Hydrocracking generally increased the nitrogen content of the final products compared with the original feed (7). This can be explained by the contribution due to cleavage of nitrogen-containing structures form asphaltenes.

LIQUEFACTION AND COPROCESSING

Chemistry of Coal Liquefaction and Coprocessing

One of the possible structures of vitrinite in bituminous coal has been reported by Given (11). It includes aromatics, hydroaromatic structures, phenolic and carbonyl groups. This structure is shown in Fig. 6. It is an average structure only and the molecular units are considered to be held together by hydrogen bonds, Van der Wall's forces and some covalent bonds such as etheric oxygen and methylene groups.

Knowledge of the structures of interacting species allows the mechanism of coal dissolution to be studied. Curran (12) has suggested a free radical mechanism for coal liquefaction. According to this mechanism the weak bonds in the coal break down homolytically to produce radicals which can be stabilized by hydrogen donor species:

> coal - coal - 2 coal • coal • H-Donor coal-H

The exact mechanism of hydrogen transfer is not known. Hydrogen may be transferred from the solvent to coal, from coal to another part of the coal via the solvent (hydrogen shuttling), or from molecular hydrogen which may be present.

In coal liquefaction the H-donor solvent is normally a coal-derived liquid which is recycled and its quality is monitored constantly. The solvent quality has a detrimental effect on product yields and should be maintained during the operation. One of the factors influencing the solvent quality is the hydrogen donor ability of the solvent. In coprocessing of coal and heavy oil/bitumen the heavy oil solvent acts as both slurry forming medium and a potential hydrogen donor solvent while being simultaneously upgraded.

CANMET COPROCESSING

Coprocessing heavy oils, bitumens or petroleum residues with coal can be considered a bridge between coal liquefaction and hydrocracking. In terms of operation, coprocessing is less complicated than liquefaction because the recycle solvent is eliminated. The effort in coprocessing is focussed on achieving residuum conversion levels as high as those in hydrocracking. This would result in significant savings in feedstock costs by substituting a significant portion of heavy oil with less expensive coal.

CANMET coprocessing involves the simultaneous upgrading of coal and heavy oil in a once-through operation using a disposable iron additive. Process feasibility has been investigated using a variety of coals and heavy oils/ bitumens (8). Also, it has been demonstrated that in terms of product yields for subbituminous coals, CANMET coprocessing is superior to liquefaction and is comparable to hydrocracking (9-10).

Feedstocks

The general characteristics of solvents and coals used in coprocessing are shown in Tables 1 and 7 respectively. Feed slurries were prepared by mixing the desired amounts of coal and vacuum bottoms under atmospheric pressure in a stirred tank. A disposable additive (coal impregnated with iron sulphate) was used and the amount of additive was kept constant on a moisture and ash free (maf) slurry feed basis for all experiments. Only the result from coprocessing Cold Lake vacuum bottoms and Forestburg subbituminous coal are presented.

Process Description

Figure 7 shows a schematic of CANMET's continuous-flow coprocessing unit. The gas feed system consists of two identical pressure and flow control lines for hydrogen and carbon monoxide. The slurry feed system consists of a slurry feed tank recirculation pump and a high pressure metering pump. The slurry feed tank is mounted on a scale to monitor and record the exact amount of slurry fed to the reactor. The recirculation pump provides additional mixing action and generates the necessary net positive suction head for the metering pump. The four-headed metering piston pump delivers the slurry feed at a constant rate to the reactor. Both the reacting gases and the slurry are fed to the bottom of a 1-L stirred tank reactor. The reactor is fitted with an overflow tube through which the products flows either to an off-specification receiver or a sample receiver. The vapours from either receiver pass through a water-cooled condenser were condensible light ends are separated. The effluent gas then passes through a scrubber and an automatic high pressure let-down system. The volume of exit gas is metered before it is vented.

Both the whole slurry product and the light ends are discharged directly into pressurized vessels which are weighed before depressurization and product workup.

EFFECT OF PROCESS SEVERITY ON COPROCESSING

Product Yields

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Figure 8 shows the distillate (C_5 to -525°C), residue (+525°C) and hydrocarbon gas yields as a function of pitch conversion* which is a measure of severity. The linear increase of distillate yield is accompanied by an equivalent linear decrease in the residue yield. The increase in the gas yield with severity is relatively small indicating that at the highest level of severity most of the hydrocracking resulted in the production of liquid rather than gaseous products.

Compound-Type Distribution

Compound-type separation was carried out on fraction boiling between 205-525°C according to the schematic shown in Fig. 4. Similar to CANMET hydrocracking, increasing severity resulted in a sharp increase in saturates and smaller increases in mono-, di- and poly-aromatics and also polar compounds (Fig. 9).

Effect of Coal Concentration on Coprocessing

Distillate yields

The effects of coal concentration on distillate yields is shown in Fig. 10. At low coal concentration there appears to be a synergism between coal and bitumen, i.e., higher distillation yields (approximately 9 wt %) were found over operating with no coal. For the range of coal concentrations of interest in coprocessing, distillate yields remain constant and equal to those for the no coal case and seem to drop for higher coal concentrations.

Compound-type distribution

The effect of coal concentration on compound-type distribution is shown in Fig. 11. Increasing coal concentration from 0 wt % to 25 wt % (maf)

*pitch conversion = $\frac{\text{maf}(+525^{\circ}\text{C}) \text{ in } - \text{ maf}(+525^{\circ}\text{C}) \text{ out}}{\text{maf}(+525^{\circ}\text{C}) \text{ in}}$

did not change significantly the product characteristics of distillates boiling between 205-525°C. The aromaticity of distillates also remained relatively unchanged as coal concentration increased.

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CONCLUSIONS

The primary upgrading of low-grade fossil fuels such as coal, bitumen, heavy oil and residues by CANMET hydrocracking and coprocessing produces large

quantities of transportation and home heating fuels of higher quality than existing commercial coking processes. Although upgrading by the hydrogenation routes increases the H/C ratio of the hydrocarbons and reduces the concentration of heteroatoms such as S, N, and O, the products of primary upgrading require additional hydrotreatment in order to meet specification fuel requirements.

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		Athabasca (+524°C)	Cold Lake (+454°C)	Boscan (+343°C)	Canadian Blend	Arabian Light (+549°C)	Lloydminster (+343°C)	Laguna (+454°C)
Specific Gravity,	15/15°C	1.073	1.038	1.016	0.989	1.023	1.006	1.024
Gravity,	•API	0.37	4.8	7.77	11.57	6.82	9.16	6.7
Sulphur,	wt %	6.20	5.82	5.73	1.37	4.30	4.49	3.43
Carbon,	wt %	83.25	82.90	82.44	86.98	84.91	83.80	85.01
Hydrogen,	wt %	9.45	9.96	10.36	11.37	9.87	10.29	11.03
Nitrogen,	wt %	0.82	0.68	0.80	0.45	0.21	0.67	0.61
Ash,	wt %	1.08	0.05	0.24	0.034	0.035	0.035	0.12
C.C.R.,	wt %	27.5	19.8	16.7	15.9	21.3	14.0	18.4
Metals ¹ ,	թթա	12512	357	1311	90	212	225	655
Viscosity at 99°C,	P1	_3	3.63	0.585	0.303	1.24	0.342	-
Pentane Insolubles,	wt %	31.4	22.7	22.3	11.8	17.8	17.6	19.6
Toluene Insolubles,	wt %	0.86	0.07	0.09	1.74	0.04	0.06	trace
Pitch (+524°C),	wt %	98.5	85.10	66.7	90.0	98.7	58.2	81.6
H/C at. ratio		1.36	1.44	1.51	1.57	1.39	1.47	1.56

TABLE 1- Characteristic properties of some processed feedstocks

1 V, Ni, Fe
2 In addition this feed contained: Al - 0.16%, Si - 0.22%, other metals - 682 ppm
3 Did not flow into viscometer at 149°C

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COMPOUND-TYPE DISTRIBUTION (WT %) IN DEASPHALTENED RESIDUES BOILING HIGER THAN 350 °C.

-	COMPOUND-TYPE							
SAMPLE	SATURATES	MONO- AROMATICS	DI- Aromatics	POLY AROMATICS	POLAR Compounds	BASIC Material		
ATHABASCA	19.4	12.9	8.1	23.2	21.7	6.5		
Cold Lake	20.0	11.9	8.9	25.1	23.5	6.9		
LLOYDMINSTER	20.3	6.9	9.6	13.4	37.7	8.2		
MEDICINE RIVE	R 56.0	14.7	8.3	7.6	11.1	2.1		

QUANTITATIVE DISTRIBUTION OF AROMATIC RING CLASSES (WT % OF THE TOTAL PAH FRACTION IN DEASPHALTENED RESIDUE >350 °C)

	ATHABASCA	cold lake	LLOYDMINSTER	MEDICINE RIVER	Processed Athabasca
3-RING	37	40	48	49	52
4-RING	21	23	23	25	27
>4-RING	25	21	19	15	13
retained on colum	17 N	16	10	11	8

COMPONENTS IDENTIFIED IN POLYNUCLEAR AROMATIC HYDROCARBON FRACTION OF COLD LAKE HYDROCRACKED PRODUCT.

COMPOUND	SAMPLES CONCENTRATION (PPM) ¹
PHENANTHRENE	84.29
DIETHYLFLUORENE	52.61
2-METHYLPHENANTHRENE	119.67
1-METHYLPHENANTHRENE	54.72
3, 6-DIMETHYLPHENANTHRENE	50.43
FLUOROANTHENE	65.13
TRINETHYLFLUORENE	54.28
PYRENE	232.74
ETHYLMETHYLDIBENZOTHIOPHENE	61.66

TABLE 4 (CONT.)

COMPOUND	SAMPLES CONCENTRATION (PPM) 1
BENZO (a) FLUORENE	56.92
4-METHYLPYRENE	176.93
METHYLPHENYLNAPHTHALENE	93.48
2,7-DIMETHYLPYRENE	62.36
BENZO(b)NAPHTHO(2,1d)THIOPHENE	106.92
TRIMETHYLPYRENE	56.06
METHYLBENZONAPHTHOTHIOPHENE	51.26
METHYLBENZO (a) ANTHRACENE	86.27

(1) PNA WAS MEASURED IN MICROGRAMS PER GRAM OF SAMPLE ANALYSED BY G.C.

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SULPHUR-TYPE DISTIBUTION IN NAPHTHA PRODUCT OBTAINED IN HYDROCRACKING OF ATHABASCA BITUMEN.

SAMPI F	SULPHUR (WT %)					
	1	2	3	4		
MERCAPTANS	0.052	0.067	0.056	0.010		
ALIPHATIC SULPHIDES	0.64	0.52	0.43	0.21		
AROMATIC SULPHIDES	0.28	0.28	0.26	0.14		
RESIDUAL SULPHUR	0.83	0.92	0.82	0.51		
total sulphur	1.80	1.79	1.57	0.87		

NITROGEN CONTENT OF THE NITROGENOUS COMPOUND-TYPE CONCENTRATES IN ATHABASCA BITUMEN AND HYDROCRACKED PRODUCTS(a)

FRACTION		NI	TROGEN (VT %)	7/) 3 4 0.23 1.54 3.72 6.22			
	FEED	1	2	3	4			
NEUTRAL	0.49	0.72	0.68	0.23	1.54			
ACIDIC	3.67	6.36	6.33	8.72	6.22			
BASIC	3.95	5.88	5.90	5.07	7.81			
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(a) DEASPHALTENED +200 °C FRACTION

Coal	Bienfait	Coronach	Hat Creek A	Hat Creek B	Forestburg	Highvale	Lingan	Illinois #6
Rank	lignite	lignite	aubbituminoua	subbitumineus	aubbituminous	subbituminous	HV bituminous	HV bituminous
Hoisture	15.30	6.39	18.52	16.50	19.11	16.95	2.86	2.39
Ash	6.87	13.41	42.52	32.41	7.67	11.08	6.83	10.52
Analysis on dry ash-free								
basis (wt \$)								
Carbon	71.04	68.29	61.65	69.45	71.65	73.14	81.10	79.04
Hydrogen	4.82	4.70	4.44	5.12	4.33	3.83	5.15	5.48
Sulphur	0.72	0.98	1.44	0.86	0.56	0.18	1.87	3.57
Mitrogen	1.25	0.92	1.65	1.66	1.82	0.97	1.74	1.87
Oxygen (by difference)	22.17	· 25.11	29.50	22.91	21.59	21.86	10.14	10.04
Volatile matter	47.52	43.37	57.88	49.99	48.53	41.46	37.37	44.74
Petrographie analysis on								
"as-received" basis								
Vitrinites	85.2	78.6	74.2	74.2	68.4	75.0	81.0	89.0
Liptinites	5.6	1.4	2.9	2.2	4.0	4.6	6.0	5.0
Inertinites	6.6	19.6	5.4	4.6	5.4	18.5	10.0	8.5
Hean reflectance	0.35	0.23	0.42	0.35	0.41	0,44	0.85	0.55
Heating value								
MJ/Kg	21.45	20.87	11.79	13.24	19.88	20.27	32.08	28.75
Btu/15	9221	8998	5071	5692	8543	8716	13792	12356
H/C atomic ratio	0.81	0.83	0.56	0.89	0.73	0.63	0.76	0.83

TABLE 7- Characteristics of coal feedstocks

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Fig. 1 - CANMET unit-simplified schematic

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Fig. 2 - Average structral model of the fractions of Athabasca bitumen (a) saturate fraction; (b) resin fraction; (c) aromatic fraction; (d) asphaltene (1)



Fig. 4 - Separation schematic flowsheet

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Fig. 3 - Average structure model of the vacuum distillate and the vacuum residue of Athabasca bitumen; (a) vacuum distillate; (b) saturate fraction of vacuum residue; (c) aromatic fraction of vacuum residue; (d) resin fra fraction of vacuum residue (1)



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Fig. 6 - Representation of idealized molecular structure of vitrinite in bituminous coal (11)

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Fig. 7 - CANMET continuous-flow coprocessing unit

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Fig. 10 - Effect of feed coal concentration on distillate yields

