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CHARACTERIZATION OF COAL LIQUIDS

Intermediate Report

Hydrocracking Upgraded Solvent Refined Coal (SRC)

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November 1976

Work Performed Under Contract No. EX-76-C-01-2010

UOP, Incorporated
Des Plaines, Illinois

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Hydrocracking Upgraded Solvent Refined Coal (SRC)

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HYDROCRACKING UPGRADED SOLVENT REFINED COAL (SRC)

ABSTRACT

In the hydrotreating processability study of Solvent Refined Coal (SRC) in addition to studying the effects of various variables, a preparative hydrotreating run was made to produce 4.5-gallons of upgraded SRC for use as feedstock in a hydrocracking study. This charge stock serves as the last of eight ERDA coal liquids to be characterized as to processability by conventional petroleum refining technology.

The upgraded SRC, containing 9.8% C_7-insolubles and 70.1% 650°F$^+$ bottoms, was processed over a commercial hydrocracking catalyst at four sets of conditions, including a catalyst activity check at startup conditions at the conclusion of the run. At base space velocity, 10°C above base temperature, and 200 psig above base pressure, conversion, defined as percentage disappearance of 650°F$^+$ bottoms, was only 11.8%. This low yield of 650°F$^-$ distillate is attributed to the presence of high molecular weight asphaltic materials.

HYDROCRACKING UPGRADED SOLVENT REFINED COAL (SRC)

INTRODUCTION

It was reported in ERDA Report FE-2010-06 (Upgrading Solvent Refined Coal) that in addition to two continuous hydrotreating runs made to study the effect of changes in operating conditions, a third run of extended duration was made at a single set of conditions. The object was to prepare 4.5 gallons of upgraded SRC for use as feedstock in hydrocracking studies. Details of this run were included in the above report. This feedstock serves as the last of eight ERDA coal liquids to be characterized under Contract E(49-18)2010.

This report covers Processability Evaluation and Routine Analyses as required by Tasks 1.0 and 1.1 of the Contract. Bench scale hydrocracking was run at four sets of conditions (two temperatures and two space velocities), including a catalyst activity check at the startup conditions at the conclusion of the run.

EQUIPMENT

Hydrocracking studies were carried out in UOP Research Plant 505, previously described (FE-2010-01, 02). The reactor was loaded with 150 ml of a commercial hydrocracking catalyst comprising Group VI and Group VIII metals on silica alumina.

CHARGE STOCK

The upgraded SRC was produced in Plant 638H, Run 4, as described in an earlier report (FE-2010-06). Table 1 is a summary of a selected overall material balance made for the purpose of obtaining product distribution data. The table shows that the upgraded SRC represented 77.5 wt.% of the SRC. Inspections of the upgraded SRC, as well as the SRC, are given in Table 2.

RESULTS AND DISCUSSION

Hydrocracking studies were carried out on a bench scale continuous unit (Plant 505, Run 827). Commercial hydrocracking conditions which may be categorized as "relatively severe" were used.

Plant products included gas, stripper overhead, and stripper bottoms. The last two were combined to provide samples for analyses and for obtaining vacuum distillation data. The composite product is referred to as liquid product in this report.

Product distribution was obtained from measurement and inspection of all effluent streams. Hydrogen consumption values were obtained in the manner described in Report FE-2010-03.

Charge stock for hydrocracking studies was the total upgraded SRC, 3392-69 (Table 2). Experiments were conducted at four sets of hydrocracking conditions. Results are summarized in Tables 3 and 4. Table 3 shows that at base space velocity, 10°C above base temperature, and 200 psig above base pressure (Period 2), conversion to 650°F$^-$ distillate was 11.8 vol.% and C_7-insolubles were reduced to 5.36% from 9.75% in the feed. Raising the temperature 10°C (Period 4) at otherwise identical conditions did not improve conversion or lower the C_7-insoluble content in the liquid product. This was the first

indication of catalyst deactivation. At the latter conditions, lowering the space velocity to 0.66 x base space velocity (Period 6) resulted in increasing the conversion to 650°F⁻ distillate slightly to 10.9% and decreasing the C_7-insolubles to 5.86%. When the temperature was lowered to 10°C above base temperature (Period 8), while other processing variables remained unchanged, conversion to 650°F⁻ distillate decreased to 6.0% while C_7-insolubles content increased to 6.57%. The run was then concluded with a catalyst activity check (Period 10) at the initial conditions. Results showed that after 119 hours of processing, conversion to 650°F⁻ distillate decreased from 11.8 vol.% to 2.0 vol.%, the product hydrogen content dropped from 9.32% to 8.83, the product nitrogen content increased from 0.153% to 0.377%, and the API gravity decreased from 5.0 to 3.2.

Table 3 also indicates that the API gravity of the liquid products were all lower than that of the feed. This is contrary to experience with catalytic hydrocracking of upgraded Synthoil (FE-2010-05, Tables 5 and 9). The lowering of API gravity in the present case appears to reflect the proportionately greater gas and light ends in the converted SRC. Although these are included in the product distribution (Table 4), they do not contribute to raising the API gravity of the liquid.

Product distribution and hydrogen consumption data are tabulated in Table 4. The table shows that hydrogen consumption during Period 2 was 519 SCF/B, and this dropped to 248 SCF/B during the final catalyst activity check (Period 10).

Data obtained from this study clearly show that, as in hydrocracking upgrading Synthoil (FE-2010-05), because of the presence of high molecular weight asphaltic materials, conversion to 650°F⁻ distillate was very low.

Table 1

Upgrading SRC

Product Distribution

Plant 638H, Run 4

Period 7

Product Distribution, wt.% of SRC Feed	
Stripper Bottoms[a]	77.5
Light Product	15.1
C_5 and C_6 in Plant Gas	2.9
Gas (C_1-C_4)	3.2
H_2O	3.4
H_2S	0.8
NH_3	2.1
Total	105.0
H_2 Consumption, wt.% of Feed	5.0
H_2 Consumption, SCF/B	3791

(a) Designated as upgraded SRC

Table 2

Inspection of SRC Samples

Sample Designation	SRC	Upgraded SRC
Sample No.	3392-1	3392-69
°API @ 60°F	-13.7	9.6
Sp. Gr. @ 60°F	1.2012	1.0028
Distillation (ASTM D-1160)		
IBP, °F	648	433
5%	834	555
10%	905	600
15%	951	
20%		660
30%		718
40%		780
50%		850
60%		940
65%		1000
% Over	15.0	65.0
% Bottoms	85.0	35.0
C_7-insol., %	89.4	9.76
Benzene Insol., %	34.7	1.68
Conradson Carbon, %	55.85	16.31
Aromatics, Wt %		91.7
Ash, %	0.11	0.001
Ultimate, %		
H	5.63	8.76
C	84.44	90.85
S	0.78	0.02
N	2.12	0.548
O	3.71	0.20
Molecular Weight, average	622	360
Pour Point, °F		+55
Viscosity, 210°F, cst		32.69
Viscosity, 250°F, cst		14.43

Table 3

Hydrocracking Upgraded SRC

Trickle Bed Operation, 150 ml Catalyst

Run 827

Period No.	Feed 3392-69	Liquid Product 2	4	6	8	10
Hours on Stream		20-28	48-56	85-95	161-131	141-147
LHSV/LHSV (base)		1.0	1.0	0.66	0.66	1.0
T-T (base), °C		10	20	20	10	10
P-P (base), psig		200	200	200	200	200
C_7-insolubles, %	9.79	5.36	6.13	5.86	6.47	7.34
Sp. Gr., @ 60°F	1.0028	1.0366	1.0435	1.0382	1.0505	1.0505
°API, @ 60°F	9.6	5.0	4.1	4.8	3.2	3.2
650°F+ Bottoms, Vol. %	70.10	61.82	65.46	62.43	65.89	68.68
Conversion to 650°F, Vol. %		11.8	6.6	10.9	6.0	2.0
Ultimate, %						
H	8.76	9.32	9.11	8.99	8.99	8.83
C	90.85	90.01	89.80	89.69	90.77	90.01
S	< 0.02	< 0.02	< 0.02	< 0.02	< 0.02	
N	0.548	0.153	0.304	0.269	0.349	0.377
O	0.20	0.11	0.16	0.10	0.10	0.13

Table 4

Product Distribution

Hydrocracking Upgraded SRC

Run 827

Period No.	2		4		6		8		10	
Hours on Stream	20-28		48-56		85-95		161-131		141-147	
LHSV/LHSV (base)	1.0		1.0		0.66		0.66		1.0	
T-T (base) °C	10		20		20		10		10	
P-P (base), psig	200		200		200		200		200	
Product Distribution	Wt.%	Vol.%	Wt.%	Vol.%	Wt.%	Vol.%	Wt.%	Vol.%	Wt.%	Vol.%
C_1-C_4 Fraction	1.09		1.41		2.44		1.76		1.44	
C_5-C_6 Fraction	0.81	1.26	0.50	0.78	1.27	1.97	1.49	2.32	0.69	1.08
C_7-390°F Fraction	3.87	4.72	1.29	1.57	3.98	4.86	2.15	2.57	2.55	2.85
390°-650°F Fraction	27.56	29.14	26.01	27.52	24.92	26.39	23.22	24.59	20.34	21.23
650°F$^+$ Bottoms	67.51	61.82	71.49	65.46	68.07	62.43	72.02	65.89	75.36	68.68
Total	100.84	96.94	100.70	95.33	100.68	95.65	100.64	95.37	100.38	93.84
Hydrogen Consumption, SCF/B	519		440		421		402		248	