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CATALYTIC CRACKING OF HYDROTREATED CONVENTIONAL AND SYNTHETIC FEEDSTOCKS

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ABSTRACT

Five gas oils from conventional and synthetic crudes were hydro-treated over sulphided Ni/Mo and Ni/W catalysts at 343°C (650°F), pressures ranging from 2.76 to 13.78 MPa (400 to 2000 psi) and LHSV of 1 or 2 h⁻¹. The treated products were then catalytically cracked in a microactivity test (MAT) unit to estimate FCC yields.

The results indicated that the gasoline yields could be increased by as much as 20-38% depending on the feedstocks and the hydrogen consumptions during hydrotreatment. In general, coker gas oil from conventional crude showed the best result followed by the virgin gas oils produced from conventional crude and heavy oil. The synthetic gas oils were more difficult to hydrotreat, consumed less hydrogen and thus produced less additional gasoline. Also, for all severely hydrotreated feeds, coke make was reduced significantly (17 to 43%).

Hydrotreatment with the Ni/Mo catalyst at 13.78 MPa, 2 LHSV and 343°C produced the best MAT feeds which gave highest gasoline yield and lowest coke make. As well, the elimination of sulphur, nitrogen and Conradson carbon was the greatest at these conditions.

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CRAQUAGE CATALYTIQUE SUR LES CHARGES
D'ALIMENTATION CONVENTIONNELS ET SYNTHÉTIQUES

par

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RÉSUMÉ

Cinq gazoles tirés de pétrole brut classique et synthétique ont été hydrotraités sur des catalyseurs Ni/Mo et Ni/W sulfurés, à une température de 343°C (650°F), à des pressions variant de 2,76 à 13,78 MPa (400 à 2000 psi) et à un VSH de 1 ou 2 h⁻¹. Les produits traités ainsi ont été soumis à un craquage catalytique dans un appareillage d'analyse de microactivité pour évaluer le rendement en craquage catalytique fluidisé.

Les résultats ont démontré que les gazoles traités ont accru considérablement le rendement en essence (20-38 % de plus, dépendant des types de gazole et de la consommation en hydrogène). En général, le gazole à partir de la cokéfaction a été le meilleur, suivi par le gazole classique et celui dérivé de l'huile lourde. L'hydrotraitement des gazoles synthétiques a été beaucoup plus difficile et a consommé moins d'hydrogène et par conséquent a produit moins d'essence additionnelle. De même, la production de coke à partir de toutes les charges d'alimentation très hydrotraitées a été considérablement réduite (17 à 43 %).

L'hydrotraitement avec le catalyseur Ni/Mo à 13,78 MPa, 2 VSH et une température de 343°C a permis d'obtenir les charges d'alimentation qui donnaient le rendement le plus élevé en essence. L'élimination du soufre, de l'azote et du carbone Conradson était également supérieure à ces conditions.

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INTRODUCTION

Faced with the fast and irreversible depletion of high quality conventional crudes, petroleum refiners are forced to utilize heavier feedstocks worldwide. In Canada, the greatest proven reserves of hydrocarbons are the heavy oil and tar sand deposits of Western Canada. The utilization of these vast resources can affect both the national trade balance and the security of energy supply. Two commercial plants have been in operation for years to produce synthetic crudes from oil sand bitumens based principally on carbon rejection technology. A new CANMET process (1) for hydrocracking bitumens, heavy oils and resids has been developed and a 5000 b/d demonstration plant integrated into an existing refinery has been in operation for some time. Due to the incompleteness of biogenesis, the bitumens and heavy oils are hydrogen-deficient (low H/C ratio) resulting in higher molecular weight. Also, they have high concentrations of impurities such as N, S and metals. These immature feedstocks require primary upgrading and present problems to the subsequent processing in a refinery. This is particularly true in the fluid catalytic cracking unit (FCCU) where the gas oils and resids are cracked. The FCC feeds derived from bitumens and heavy oils are highly aromatic and refractory, and contain more impurities than those from conventional crudes. Cracking of these materials is more difficult resulting in inferior product quality. Also, the catalyst deteriorates faster due to poisoning by metals (such as Ni, V, Cu and Fe), coke and basic nitrogen which deactivates acid sites of the catalyst. In addition, the exhaust gas, rich in SO_x and NO_x , from the regenerator imposes corrosion and environmental problems. Since the FCCU is the major producer of gasoline, accounting for about 35% of the gasoline sold in North America and Europe (2), the trend of charging more nonconventional feedstocks to FCCU has caused general concern among refiners; thus research is being conducted to find better ways to process these materials. It has been reported that hydrotreating of heavy feedstocks prior to FCC operation generally can increase conversion yield, improve product quality and prolong catalyst life (3). The objectives of this study were: 1) to evaluate the effect of hydrotreating on the quality of conventional and synthetic gas oils; 2) to determine the potential FCC product yields of raw and treated gas oils in a microactivity test (MAT) unit; 3) to assess the benefits of hydrotreating-cracking combination over direct cracking.

EXPERIMENTAL

Five different gas oils (GO) from commercial plants were used:

- Sweet GO - virgin gas oil from sweet (low S) conventional crude;
 Coker GO - raw coker gas oil from conventional crude;
 Sour GO - virgin gas oil from Lloydminster sour blend (high S heavy oil);
 Synthetic GO A - gas oil derived from Athabasca tar sand bitumen upgraded in a delayed coker;
 Synthetic GO B - gas oil derived from Athabasca tar sand bitumen upgraded in a fluid coker.

Prior to hydrotreating, the sweet gas oil was fractionated to bring the boiling point range in line with the other gas oils. Hydrotreating was conducted in a downflow fixed bed reactor with a once-through gas design.

Four reaction conditions and two commercial catalysts were used:

| <u>Conditions</u> | <u>Temperature (°C)</u> | <u>Pressure (MPa)</u> | <u>Weight hourly space velocity (WHSV) (h⁻¹)</u> |
|-------------------|-----------------------------|---------------------------|---|
| 1 | 343 | 2.76 | 2 |
| 2 | 343 | 5.51 | 2 |
| 3 | 343 | 13.78 | 2 |
| 4 | 343 | 5.51 | 1 |

| <u>Catalyst</u> | <u>Quantity (mL)</u> | <u>Description</u> |
|-----------------|----------------------|---|
| Katalco NT-550 | 150 | 3.9% Ni, 15.9% W, Al ₂ O ₃ -based, surface area 230 m ² /g, pore volume 0.5 mL/g, sulphided |
| Katalco NM-506 | 150 | 5.3% Ni, 18.0% Mo, Al ₂ O ₃ -based, surface area 220 m ² /g, pore volume 0.39 mL/g, sulphided. |

Hydrotreating of the five feedstocks yielded 40 products which were then catalytically cracked in a MAT unit (4) loaded with 4 g equilibrium metal-tolerant catalyst Davison DA-250 (455 ppm Ni, 1951 ppm V, 35.9% Al_2O_3 , 0.23% C, surface area $73 \text{ m}^2/\text{g}$, pore volume 0.23 mL/g) obtained from a refinery. Cracking took place at 490°C with catalyst/oil ratio of 4 and WHSV of 9 h^{-1} . A nitrogen flow at 20 mL/min was started when oil was injected into the reactor for a total flow time of 1000 s. Both liquid and gas products were collected during cracking.

Feedstocks for hydrotreating, MAT feeds, MAT products and the spent catalysts were characterized physically and chemically using ASTM or generally accepted methods. Compositional analyses were obtained by mass spectrometry in conjunction with chromatography and/or distillation. MAT products were analyzed for H_2 , CH_4 , LPGs, gasoline (C_5 to 220°C), light cycle oil (LCO) ($220\text{-}344^\circ\text{C}$), decant oil (DO) ($344+$ $^\circ\text{C}$) and coke by mass spectrometry, GC simulated distillation and a combustion-titration technique.

RESULTS AND DISCUSSION

The primary objective of this study was to evaluate the effect of hydrotreating various gas oils on their catalytic cracking yields. It was expected that the uptake of hydrogen by the feedstocks during hydrotreating would increase the amount of precursors of gas + gasoline while reducing the precursors of decant oil + coke and the impurities in the MAT feeds. This can best be demonstrated by the changes in physical and chemical properties of the feedstocks before and after hydrotreating. Table 1 shows the results of five feedstocks hydrotreated at condition 3 using Ni/Mo catalyst NM-506. The improvement in product quality over that of the feed is evident. Compared with the feeds, the corresponding products showed that 1) they were less refractory as reflected by higher aniline point (more paraffinic), API gravity and lower Conradson carbon content, viscosity and boiling point range; 2) they contained less poisons (basic nitrogen and metals) and pollutants (sulphur and nitrogen); 3) as expected, they appeared to have more precursors of gas and gasoline and less precursors of decant oil and coke based on compositional analyses. The concentrations of precursors were estimated assuming the saturates and monoaromatics would yield gas and gasoline; the diaromatics,

two-ring aromatic sulphur and one half of the three-ring aromatic sulphur would contribute to the light cycle oil whereas the rest of the heavy components would form decant oil and coke upon cracking.

Table 1 shows that under the same condition the sour and coker gas oils were much more amenable to hydrotreating than the other three which showed the same degree of improvement. However, it should be mentioned that the sweet conventional gas oil was a comparatively superior feedstock and was unlikely to improve much. Thus, it is concluded that the two synthetic feedstocks are less treatable.

Examination of other experimental data reveals that severity 3 (343°C , 13.78 MPa , 2 h^{-1}) and catalyst NM-506 ($\text{Ni-Mo/Al}_2\text{O}_3$) gave the best hydrotreating results. Table 2 shows the compositional changes of the sour feedstock hydrotreated at various conditions. The great increase in the amount of saturates and monoaromatics especially at severity 3 and with catalyst NM-506 produced much more gas and gasoline precursors in the product at the expense of the precursors of LCO, DO and coke. It should be noted that at 343°C the major process in this hydrotreating study is the partial or complete saturation of the benzene rings and the olefins. Both the reversible reaction, i.e., dehydrogenation, and hydrocracking are not likely to occur significantly below 400°C (5). Table 2 shows that hydrogenation of the aromatics and the unsaturates is more effective at higher pressure and lower space velocity and that catalyst MN-506 is more active than NT-550.

Following quality improvement, the treated feedstocks generally show better catalytic cracking performance as reflected by the enhanced crackability, selectivity and product quality (6). The degree of improvement is usually proportional to the amount of hydrogen consumed during hydrotreating (7). Figure 1 depicts the reduction of impurities (S,N and Conradson carbon) and precursors of DO + coke, and the increase in precursor concentration of gas + gasoline in the feedstock with the hydrogen consumption. Figure 1A shows that for the sour gas oil more than 90% of the impurities could be removed after consuming about 600 SCFB of H_2 . Figure 1B demonstrates that upon catalytic cracking both conversion and gasoline yield increased linearly whereas the coke yield decreased linearly with hydrogen consumption. Since hydrogen consumption data were obtained from runs at various severities and with two different catalysts, this implies that the hydrogen consumption is the key factor

in determining the fate of the subsequent catalytic cracking of the treated feedstocks. Table 3 summarizes the hydrogen consumption data of all the test runs. It can be seen that higher pressure and lower space velocity favoured the increase in hydrogen consumption. The two synthetic gas oils, particularly synthetic GO B, consumed less hydrogen than the coker and sour gas oils. Of the two catalysts, NM-506 was generally a better hydrotreating catalyst in this study.

Figure 2 illustrates the product distribution of the key components when the raw and hydrotreated feedstocks (severity 3, catalyst NM-506) were catalytically cracked in the MAT unit. In all cases the gasoline and/or gas yields increased at the expense of light cycle oil, decant oil and coke. Among the five treated feedstocks, coker gas oil showed the biggest increase in gasoline (38.3 %) over the untreated one, followed by the sweet GO (31%), sour GO (26.4%), syncrude GO A (19.8%) and syncrude GO B (19.5%). The decrease in coke yield varied from 43.1% (coker GO) to 16.9% (syncrude GO A). It should be mentioned that on absolute scale the increase in gas yield was rather mild compared with that in gasoline yield.

The economic advantages of hydrotreating FCC feedstocks in a commercial process depends on the capital and operating costs of a hydrotreater and a hydrogen plant. Also, the amenability of the feedstock to hydrotreating, the capacity of the FCC unit and current prices of the incremental FCCU products, etc. should be considered. Therefore, the real detailed economics must be evaluated on an individual basis and is beyond the scope of this study. However, a rough estimate by considering a portion of the variables may shed some light on the benefits of this combined process. Assuming that the MAT yield reflects the commercial FCC production, and the three fractions of the liquid product can be treated as motor gasoline, heating oil (No. 2) and residual fuel oil (No. 6) at \$18.80, \$18.63 and \$14.50 per barrel respectively in December 1986 (8), then the increase in gasoline yield due to hydrotreating at the expense of light and heavy fuel oils may create a profit of about \$5 million/a for the syncrude GO B (hydrotreated at severity 3 using catalyst NM-506) which is to be cracked in a 30 000 b/d FCC unit. This can be a substantial payout on the investment for the extra facilities. This still does not consider the profit resulting from the mild increase in the gas yield, the improved quality of the cracked products, the prolonged life of the catalyst, the diminution of the pollution and corrosion problems, etc. The substantial decrease in coke yield may allow for the addition of residuum to a hydrotreated feedstock and still maintain acceptable coke levels in the FCC unit.

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Table 1 - Physical and chemical properties of raw and hydrotreated feedstocks

| Gas oil | Sweet | | | Coker | | | Sour | | | Synthetic A | | | Synthetic B | | |
|--|--------|---------|--------|--------|---------|--------|--------|---------|--------|-------------|---------|--------|-------------|---------|--------|
| | Raw | Treated | Diff. | Raw | Treated | Diff. | Raw | Treated | Diff. | Raw | Treated | Diff. | Raw | Treated | Diff. |
| Hydrotreating catalyst | NM-506 | | | NM-506 | | | NM-506 | | | NM-506 | | | NM-506 | | |
| Reaction condition | 3 | | | 3 | | | 3 | | | 3 | | | 3 | | |
| H ₂ consumption, SCFB | 481 | | | 685 | | | 628 | | | 398 | | | 226 | | |
| Gravity, °API | 27.4 | 29.5 | +2.1 | 22.3 | 28.7 | +6.4 | 21.6 | 26.9 | +5.3 | 23.3 | 25.6 | +2.3 | 20.2 | 22.2 | +2.0 |
| Aniline Pt., °C | 88.5 | 92.6 | +4.1 | 72.1 | 79.8 | +7.7 | 66.1 | 78.6 | +12.5 | 59.3 | 64.7 | +5.4 | 56.5 | 62.0 | +5.5 |
| Conradson carbon, wt % | 0.12 | 0.015 | -0.105 | 0.04 | 0.003 | -0.037 | 0.09 | 0.004 | -0.086 | 0.04 | 0.002 | -0.038 | 0.09 | 0.028 | -0.062 |
| Total nitrogen, wt % | 0.056 | 0.009 | -0.047 | 0.163 | 0.034 | -0.129 | 0.049 | 0.005 | -0.044 | 0.074 | 0.017 | -0.057 | 0.089 | 0.047 | -0.042 |
| Basic nitrogen, wt % | 0.015 | 0.005 | -0.010 | 0.053 | 0.012 | -0.041 | 0.019 | 0.002 | -0.017 | 0.024 | 0.005 | -0.019 | 0.020 | 0.012 | -0.008 |
| Total sulphur, wt % | 0.57 | 0.05 | -0.52 | 0.44 | 0.04 | -0.40 | 2.03 | 0.10 | -1.93 | 0.33 | 0.05 | -0.28 | 0.24 | 0.10 | -0.14 |
| Viscosity at 40°C, cSt | 10.48 | 9.71 | -0.77 | 11.88 | 10.84 | -1.04 | 28.89 | 19.41 | -9.48 | 12.92 | 12.02 | -0.90 | 27.13 | 25.23 | -1.90 |
| Viscosity at 100°C, cSt | 4.51 | 4.37 | -0.14 | 2.85 | 2.74 | -0.11 | 4.48 | 3.70 | -0.78 | 2.82 | 2.73 | -0.09 | 4.15 | 4.01 | -0.14 |
| <u>Metals µg/g</u> | | | | | | | | | | | | | | | |
| Ni | <0.05 | <0.05 | | <0.05 | <0.05 | | 0.05 | <0.05 | | <0.05 | <0.05 | | <0.05 | <0.05 | |
| V | <0.05 | <0.05 | | <0.05 | <0.05 | | 0.18 | <0.05 | | <0.05 | <0.05 | | <0.05 | <0.05 | |
| Fe | 0.22 | 0.11 | -0.11 | 0.8 | 0.49 | -0.31 | 2.2 | 0.28 | -1.92 | 2.1 | 2.6 | +0.5 | 0.7 | 0.35 | -0.35 |
| <u>GC distillation, °C</u> | | | | | | | | | | | | | | | |
| IBP | 241 | 242 | | 204 | 238 | | 250 | 223 | | 215 | 226 | | 196 | 214 | |
| 5% | 291 | 285 | -6 | 290 | 274 | -16 | 307 | 277 | -30 | 257 | 251 | -6 | 260 | 259 | -1 |
| 10% | 317 | 307 | -10 | 309 | 298 | -11 | 329 | 299 | -30 | 274 | 266 | -8 | 287 | 281 | -6 |
| 50% | 414 | 404 | -10 | 366 | 356 | -10 | 394 | 376 | -18 | 349 | 337 | -12 | 376 | 366 | -10 |
| 90% | 493 | 479 | -14 | 427 | 421 | -6 | 466 | 449 | -17 | 443 | 431 | -12 | 469 | 459 | -10 |
| FBP | 566 | 584 | | 493 | 528 | | 552 | 560 | | 521 | 549 | | 568 | 566 | |
| <u>Precursors</u> (estimated), wt % | | | | | | | | | | | | | | | |
| gas + gasoline | 79.1 | 90.1 | +11 | 72.2 | 87.4 | +15.2 | 65.9 | 86.7 | +20.8 | 72.0 | 85.5 | +13.5 | 70.0 | 80.3 | +10.3 |
| LCO | 10.6 | 6.6 | -4 | 14.3 | 8.8 | -5.5 | 20.4 | 8.8 | -11.6 | 17.6 | 9.8 | -7.8 | 18.3 | 12.8 | -5.5 |
| DO + Coke | 10.4 | 3.4 | -7 | 13.5 | 3.8 | -9.7 | 13.7 | 4.5 | -9.2 | 10.4 | 4.7 | -5.7 | 11.6 | 6.9 | -4.7 |

Table 2 - Compositional analyses of the raw and hydrotreated sour gas oils

| Hydro- treating | Catalyst Condition H ₂ Consumption, SCFB | NT-550 | | | | NM-506 | | | | |
|-------------------------------------|---|---------|---------|---------|---------|---------|---------|---------|---------|--|
| | | 1 | 2 | 3 | 4 | 1 | 2 | 3 | 4 | |
| | | 328 | 533 | 538 | 458 | 369 | 480 | 628 | 556 | |
| | Raw | Treated | Treated | Treated | Treated | Treated | Treated | Treated | Treated | |
| <u>Hydrocarbon type, wt %</u> | | | | | | | | | | |
| Paraffins | 14.2 | 14.8 | 17.3 | 17.2 | 16.6 | 16.8 | 17.4 | 16.6 | 16.6 | |
| Monocycloparaffins | 11.2 | 12.7 | 13.8 | 13.9 | 13.3 | 13.1 | 14.1 | 14.9 | 14.7 | |
| Condensed cycloparaffins | 22.4 | 24.7 | 27.5 | 28.0 | 26.7 | 26.2 | 28.3 | 30.4 | 28.5 | |
| Alkylbenzenes | 5.94 | 10.2 | 8.90 | 8.67 | 9.04 | 8.58 | 7.94 | 8.29 | 8.43 | |
| Benzocycloparaffins | 6.11 | 7.76 | 7.06 | 7.73 | 8.07 | 7.41 | 7.05 | 8.45 | 9.12 | |
| Benzodicycloparaffins | 6.08 | 5.85 | 5.84 | 7.19 | 7.13 | 5.80 | 6.53 | 8.13 | 8.02 | |
| Diaromatics | 15.3 | 13.3 | 10.7 | 8.85 | 10.2 | 12.1 | 9.31 | 7.21 | 8.23 | |
| Triaromatics | 5.46 | 3.89 | 3.03 | 2.26 | 2.58 | 3.75 | 2.54 | 1.36 | 1.98 | |
| Tetraaromatics | 2.74 | 1.41 | 0.82 | 0.90 | 1.08 | 1.62 | 1.24 | 0.72 | 0.81 | |
| Polyaromatics & polars | 3.32 | 2.19 | 2.63 | 2.42 | 2.62 | 1.67 | 2.72 | 1.41 | 1.39 | |
| <u>Aromatic sulphur</u> | | | | | | | | | | |
| 2-ring compounds | 3.64 | 0.92 | 0.55 | 0.75 | 0.80 | 1.16 | 0.89 | 0.81 | 0.93 | |
| 3-ring compounds | 2.96 | 2.09 | 1.73 | 1.90 | 1.71 | 1.68 | 1.72 | 1.57 | 1.23 | |
| 4-ring compounds | 0.38 | 0.11 | 0.10 | 0.14 | 0.12 | 0.04 | 0.10 | 0.13 | 0.05 | |
| Unidentified hydrocarbons | 0.32 | 0.15 | 0.05 | 0.07 | 0.09 | 0.16 | 0.21 | 0.07 | 0.02 | |
| <u>Precursors (estimated), wt %</u> | | | | | | | | | | |
| Gas + gasoline | 65.9 | 76.0 | 80.4 | 82.7 | 80.8 | 77.9 | 81.3 | 86.7 | 85.4 | |
| Light cycle oil | 20.4 | 15.2 | 12.1 | 10.6 | 11.9 | 14.1 | 11.1 | 8.81 | 9.78 | |
| Decant oil + coke | 13.7 | 8.79 | 7.50 | 6.74 | 7.35 | 8.08 | 7.67 | 4.47 | 4.86 | |

Table 3 - Hydrogen consumption in hydrotreating study

| Hydrotreating catalyst | NT-550 | | | | NM-506 | | | |
|------------------------|--------|-----|-----|-----|--------|-----|-----|-----|
| | 1 | 2 | 3 | 4 | 1 | 2 | 3 | 4 |
| Sweet gas oil | 50 | 119 | 234 | 176 | 367 | 334 | 481 | 232 |
| Coker gas oil | 398 | 420 | 506 | 453 | 356 | 441 | 685 | 606 |
| Sour gas oil | 328 | 533 | 538 | 458 | 369 | 480 | 628 | 556 |
| Synthetic gas oil A | 204 | 244 | 533 | 389 | 195 | 293 | 398 | 316 |
| Synthetic gas oil B | 5 | 399 | 157 | 158 | 148 | 396 | 226 | 245 |

FIGURE CAPTIONS

- Fig. 1 - A) Effect of hydrogen consumption on the removal of sulphur (1), total nitrogen (2) and Conradson carbon (3) in the MAT feed;
B) Effect of hydrogen consumption on the precursor concentrations of gas and gasoline (1), decant oil and coke (2) in the MAT feed, and on the catalytic cracking yields - conversion (3), gasoline (4) and coke (5). MAT feed used is sour gas oil. Open and solid symbols represent runs with catalysts NT-550 and NM-506, respectively.
- Fig. 2 - Catalytic cracking yields and product distribution of raw and hydrotreated MAT feeds.

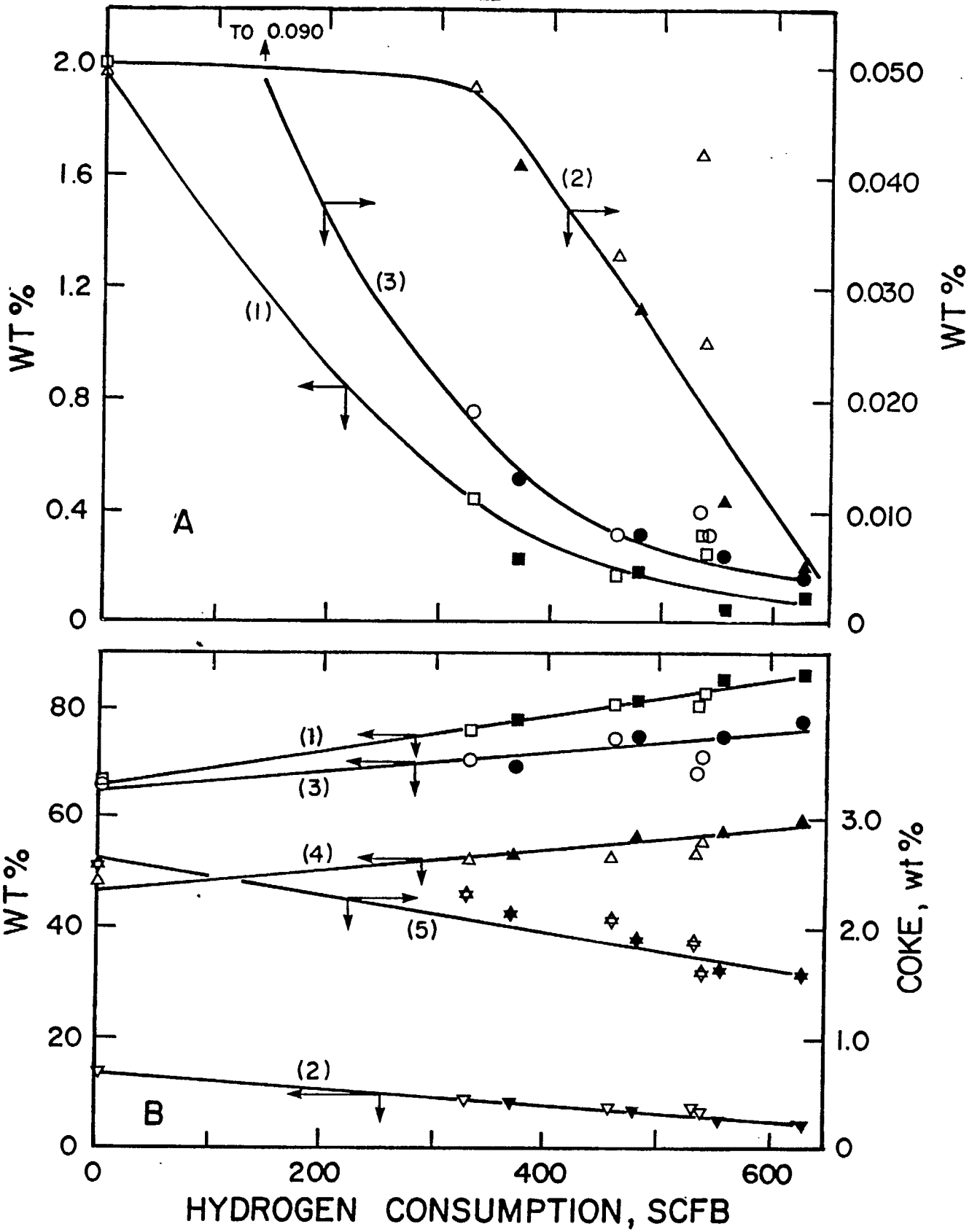


Fig. 1

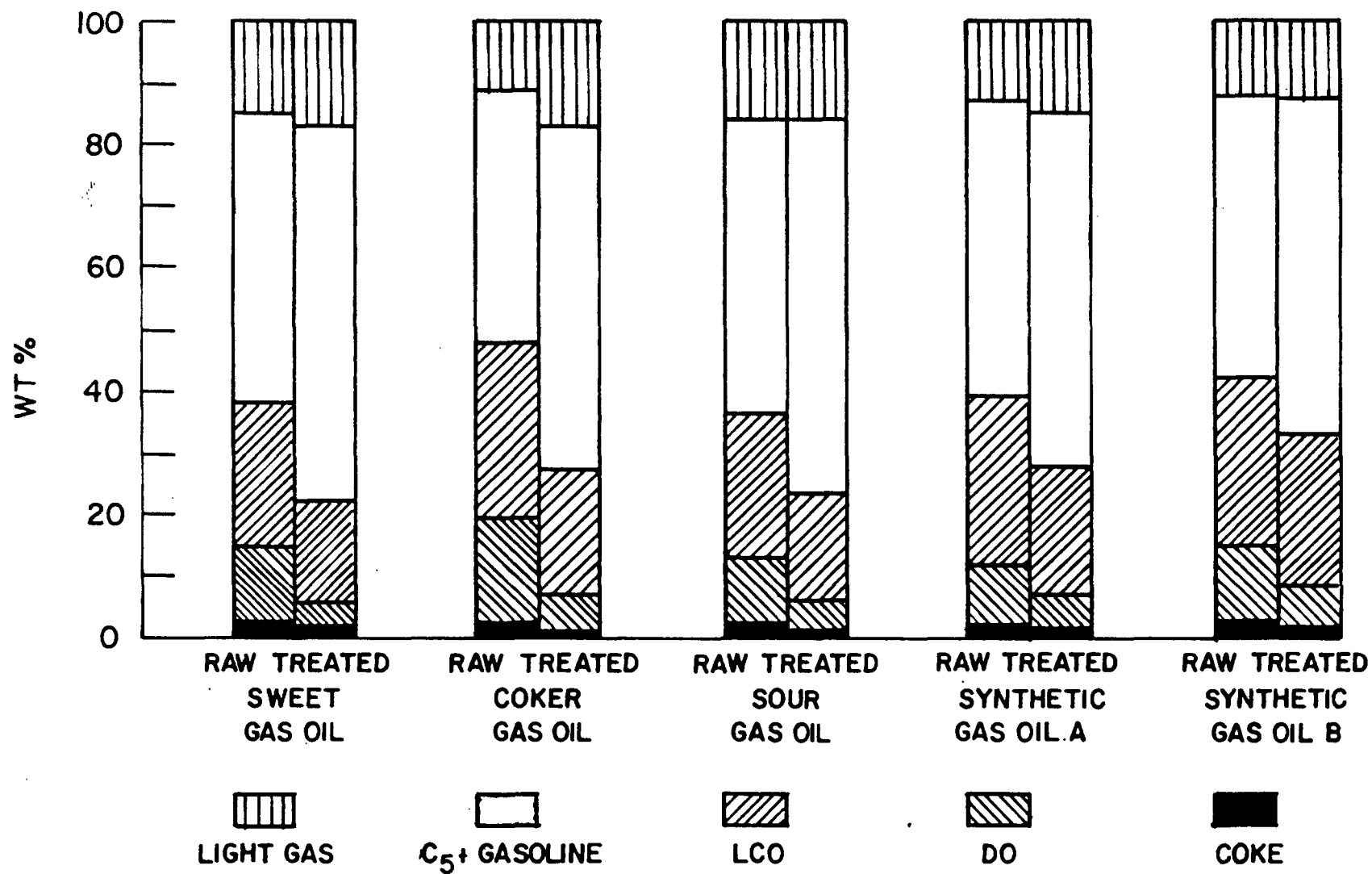


Fig. 2