COMPARATIVE STUDIES ON HYDROCRACKING WITH HYDROVISBREAKING/HYDROCRACKING COMBINATION

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ABSTRACT

A heavy vacuum residue (302.3 ppm Ni+V, 4.36% S, and 23% asphaltenes) was hydrocracked over bifunctional sulfided catalyst W-Ni-P/SiO_2-Al_2O_3, at different reaction temperatures, 350°-425°C and 120 bar hydrogen in a batch autoclave reactor. The activity of the sulfided catalyst towards hydrocracking reaction scheme alone was compared with a combination scheme (hydrovisbreaking followed by hydrocracking) in order to produce light fractions suitable for automotive fuels. The catalysts gave, at higher temperature 425°C, about 80% feedstock conversion, 5% coke make, 98% HDS and asphaltenes reduction 73%, when applying the combination scheme, compared with hydrocracking alone. These results indicated that hydrovisbreaking was a suitable alternate treatment option for heavy vacuum residue before hydrocracking process.

INTRODUCTION

In petroleum refining, hydrocracking of heavy hydrocarbons is carried out to produce high quality gasoline, jet fuel, gas oil and lubricants and to deeply remove heteroatoms. Most of hydrocracking catalysts are dual functional catalysts. They have hydrogenation–dehydrogenation function as well as acidic function. The cracking activity was controlled meanly by the support, which is acidic in nature, whereas the hydrogenation–dehydrogenation activity is due to the metals loaded on the support [Gosselink & Stork (1997)].

In hydroconversion processes, vanadium and nickel, present in the feed, are removed to a significant extent and deposited on the catalyst. The deposition of these metals caused deactivation of the employed catalyst and, if operation is continued after the metals uptake capacity of the catalyst has been exceeded, it will ultimately lead to catalyst bed plugging.

So, the need to maximize the amount of premium products from each barrel of crude or heavy residues is becoming more important. Consequently, an understanding

of the variable residue processing options and there impact on refinery yields and economics allows the refiner to select the optimum residue-processing route consistent with both the quantity and quality of the available residue and the required product distribution.

This can be overcome by selecting some upgrading methods either by addition of hydrogen, or rejecting carbon, or using a combination of both methods [**Galiasso *et al.,* (2002); Montanari *et al.,* (2002); Bearden *et al.,* (2003)**]. Generally, the high organometallic and carbon residue contents of heavier residues cause excessive catalyst deactivation when processed in hydroprocessing units.

Atemps to increase conversion of heavy oils during visbreaking will bring on sediments deposition. However, this can be changed by use of hydrovisbreaking. Among the thermal process of hydroconversion, the hydrovisbreaking differs from visbreaking in the addition of exess hydrogen in the thermal treatment.

Hydrogen in the usual hydrovisbreaking of petroleum residues or heavy oils reactions has an inhibitor effect in the condensation reactions that leads to coke formation. Therefore, hydrovisbreaking is a possible alternate option before hydrocracking process, named a combination scheme. In this study, hydrocracking and a combination of hydrovisbreaking followed by hydrocracking of heavy vacuum residue were studied in order to produce light or middle distillates within the environmental specifications.

EXPERIMENTAL

1.Feedstock:

The feedstock used in this study was heavy vacuum residue (HVR), the main Characteristics of which are shown in Table (1).

Table (1): Properties of heavy vacuum residue (> 565°C) feedstock

Characteristic	
Specific gravity at 70°C	0.993
API-gravity	18.6
Total sulfur, wt %	4.36
Conradson Carbon Residue (CCR), wt %	18.7
Nickel content, wppm	138.0
Vanadium content, wppm	164.3
Asphaltenes (n-C_7 insoluble), wt %	13.0
C/H, wt %	84.8/10.2
SARA, wt %:-	
Saturates	24.2
Aromatics	35.7
Resins	17.7
Asphaltic resins	9.4

2.Catalyst used:

A conventional mesporous hydrocracking catalyst was used in the catalytic conversion experiments. The catalyst was composed of synthetic silica alumina support (high alumina) together with metal oxides of tungsten, nickel and phosphorous. The catalyst was composed of 15 wt %, 4 wt % Ni and 1.2 wt % phosphor, and had a surface area of 220 m^2g^{-1} and pore volume of 0.48 cm^3g^{-1}. The silica/alumina ratio was 3.6 and its sulfur content after sulfidation was 5 wt%.

3.Apparatus and experimental operational procedure:

The conversion run experiments were undertaken in a stainless steel 500 mL batch autoclave reactor (Parr reactor –model 7575), fitted with a a magnetic stirrer and equipped with an internal furnace providing fast heating rates and insitu water cooling system to quench quickly the reaction during the tested reaction temperature increased.

Prior to the activity tests (i.e. hydrocracking), a sufficient quantity of the catalyst for all the experiments was first presulfided (in the autoclave) at the following conditions: 15 bar, initial hydrogen pressure, 350°C reaction temperature and 4 h reaction period. The liquid feed used in the sulfiding experiment was light gas oil containing 8% (of the catalyst) dimethyldisulfide (DMDS). After sulfidation, the catalyst was separated, washed with light solvent, dried at 100°C and kept in desiccator until used.

The sulfided catalyst ($WNi-P/SiO_2-Al_2O_3$) was reloaded into the autoclave, in the ratio of 1/10 catalyst-to-feed, for conducting the catalytic experiments under study. Amount of 350 g of feed and 35 g of the investigated catalyst was loaded and tightly closed. After repeat replacement of the air in the reaction system with hydrogen gas before the test, hydrogen was then introduced into the reactor at the beginning of the experiment from a hydrogen vessel. The reactor was pressurized to 80 bar and held for around 30 min to make sure that the system was tight and can withhold this amount of pressure. After passing the pressure leakage test, the reactor pressure was adjusted to the working initial pressure (50-60 bar), and the heating program was started since the mixture of feedstock (heavy vacuum residue) and catalyst solidifies at room temperature, the stirrer is not started immediately in order to protect it from being damage.

The mixer was started after the reactor temperature reached 150°C at which the feed-catalyst mixture was at the liquid state, and kept for 30 min, then start counting a second period of 2.5 h to reach the 3 h total time of the run. During reaction, the reaction temperature and final hydrogen pressure (120 bar) were remained constant, but when reaction temperature reached over, the furnace was stopped automatically by internal water cooling quenching system. After the reaction had been completed, reaction was immediately stopped by switching off the heating program and mixture rotation. The system was allowed to cool down to room temperature and the reached reaction pressure was recorded, and then purged out the autoclave. A gas product sample was collected, during purging, using a gas balloon for gas chromatographic analysis. The volume of the gas product was measured by a wet-gas meter after passing through a base solution, in order to reduce corrosive H_2S and NH_3 gases. All gas and liquid products were therefore recovered, weighed. Gas samples were analyzed

chromatographically, while liquid samples were analyzed using the recommended standard methods.

Solid spent catalyst after purging the liquid products was collected and washed with toluene under reflux to remove adsorbed residual oil whereas solids obtained (spent catalyst and coke) were dried at 100°C. Dried spent catalyst was regenerated by oxidation under air atmosphere at 500°C during 4 h, and the amount of coke was determined as the weight difference among solid after and before calcinations.

RESULTS AND DISCUSSION

1.Hydrocracking process:

1.1.Influence of temperature on hydrocracking process:

Fig. (1) shows the effect of process temperatures on the conversion of (HVR) via hydrocracking reactions, under 120 bar H_2 pressure and 3h reaction time, for total liquid yield, hydrocarbon gases, and coke formation. This figure indicates that the hydrocarbon gas and coke production was increased from 3.9 to 8.1 wt % and from 1.6 to 5.6 wt % respectively, with the rise of process temperature from 350° to 425°C, while total liquid yield decreases from 94.5 to 86.5 wt %. This was attributed to the following reasons:

The heavy vacuum residue (HVR) hydrocracking process was operated to maximize the yield of fractions lighter than HVR, and to subject the hydrocracking products to hydrofining, under safety process conditions. The principal function of the employed sulfided catalyst, $WNi-P/SiO_2-Al_2O_3$, in the studied experiments is to eliminate the condensation reactions via hydrogenation of highly unsaturated compounds (asphaltenes) produced during the conversion of HVR material.

The condensation reactions are responsible for the formation of coke in the reactor. Another important function of that catalyst was to promote the hydrofining reactions of hydrocracking products inclusive principally of hydrodesulfurization (HDS) reactions.

The employed $WNi-P/SiO_2-Al_2O_3$ system was a bi-functional catalyst which catalyzes both cracking and hydrogenation reactions. The fission function in this catalyst was provided by its support; i.e. $SiO_2-Al_2O_3$. Its splitting activity was dependent on activity of its acid sites since they control the reaction, which follows the ionic mechanism; i.e. for example cracking of C-C bonds [Usui *et al.*, (2004)]. It was found that the conversion of HVR was independent on catalyst concentrations, which suggest that HVR cracking reactions are predominantly thermal C-C cleavage reactions and hydrogenation reactions [**Morawski & Mosio-Mosiewski, (2006); Ali, (2007)**].

The support employed for this catalyst was highly acidic and its splitting capacity was higher. The hydrogenation function is the duty of transition metals W-Ni-P dispersed over the supporting surface, while phosphor as a catalytic activity promoter. The hydrogenation reactions catalyzed by those metals follow the free-radical mechanism.
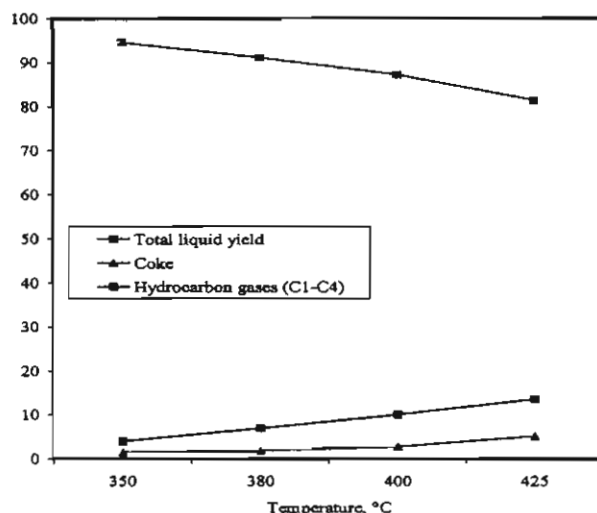
Fig.(1): Effect of process temperature on hydrocracking of HVR for total liquid yield, hydrocarbon gases and coke production.

1.2.Influence of process temperature on the products composition:

As the primary objective of the experimental conditions was conversion of heaviest and large molecules to lighter distillates, the effect of process temperatures on HVR conversion by hydrocracking for light distillates production were obtained in Fig.(2).

It is essential that the efficiency of any catalyst used in heavy vacuum residue hydrocracking be evaluated in terms of either distillate yield or conversion. As shown in Fig.(2), the distillate yields increased with the reaction temperature.

When the reaction temperature increased, the heaviest fraction (350°C+) after separated from the total distillates was clearly declined. Within the studied scope, the most light weight fractions were growing up.

The weight of the gasoline fraction (C_5-160°C) in the products was higher than that of gas fraction, i.e. amounts to 23.5 wt % at 350°C and then slightly increased to 28 wt % at 425°C. The effect of temperature on yielded kerosine fraction (160°-250°C) was nearly moderate, and amounts to 11.7 wt % at 350°C, then grew up a little to be 17 wt % at 425°C. The yield of light gas oil (or diesel) fraction (250°-350°C) amounts to 14 wt % at 350°C, and it then increased to 21 wt % at 425°C. On the other hand, the yield of middle distillate fractions (i.e. 160°-350°C) was increased as temperature increased, and heavy residue, i.e. boiling range above 350°C, decreased from 45.3 wt % at 350°C to 20.3 wt % at 425°C. The reason was that more residues undergo cracking to lighter fractions at the studied conditions. The residue fraction (350°+) was not of any less value than the motor fuel fractions. It can be fed to catalytic cracking plants in the refinery for production of low-sulfur fractions of motor fuels (gasoline and diesel).

It was also suitable and applicable as fuel oil in the turbine electric power stations, due to its upgraded characteristics, especially its reduced sulfur contents.
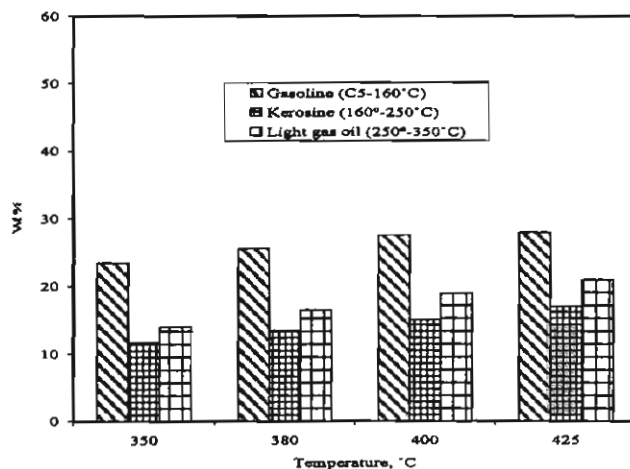

Fig. (2): Effect of process temperature on hydrocracking of HVR for distillates production.

1.3.Influence of process temperature on HVR conversion:

Fig. (3) indicates the effects of reactor (reaction) temperature on the conversion of HVR constituents such as; sulfur, conradson carbon residue (CCR), asphaltenes, beside the conversion of feedstock itself via hydrocracking reactions. It is shown that, the level of these constituents was upgraded and nearly reduced.

Nearly rectilinear increase in conversion of the HVR and its characteristics was observed through the reaction temperature range; 350°-425°C. It was assumed that at high reaction temperatures, the acidic sites on the employed catalyst also promote cracking reaction. The conversion of the feed stock itself increased from 53.1 wt % at 350°C to 74.1 wt % at 425°C. Hence, the process temperatures strongly affect the conversion of the vacuum residue. The changes of asphaltenes, CCR and sulfur have the same increase trend. The influence of temperature on the sulfur reduction, i.e. hydro-desulfurization (HDS) was much pronounced, which results from the activity characteristics of the catalyst used in the present study. Sulfur content at 350°C was found to be 0.97 wt % (i.e. conversion equals 78 %), but decreased at 380°C to 0.74 wt % (i.e. conversion equals 83%), and decreased further at 400°C to 0.54 wt % (i.e. conversion equals 88 %). Drastic decrease to 0.21 wt % was exhibited at 425°C (i.e. conversion equals 95 %).
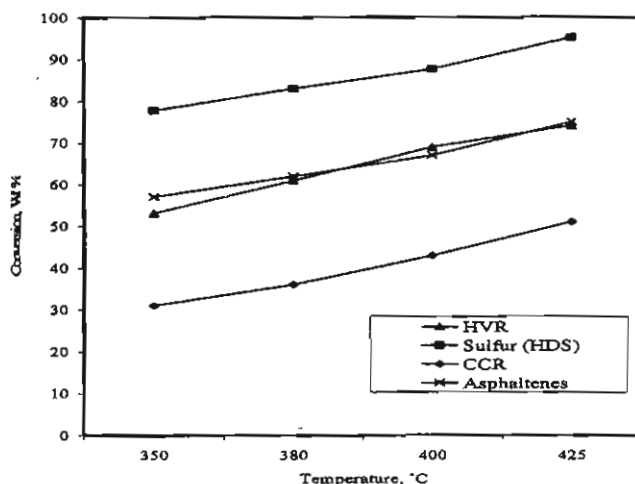
Fig. (3): Influence of process temperature on hydrocracking of HVR- conversion.

It was noticed that the high reaction pressure, 120 bars, decreased throughout the reaction period, indicating that hydrogen activity continued throughout the reaction process with the sulfided W-Ni-P/SiO_2-Al_2O_3 catalyst. Therefore, a decrease of the pressure, from 120 bars, during the tests (in the range of 80-90 bar) was observed and the final quantity of hydrogen still remains in large excess at the end of the reaction period, which also corresponds to the effects produced by coke suppression [**Matsumur et al., (2005); Danial-Fortain et al., (2009)**]. These results may be taken as eridences for the high activity of the employed catalyst W-Ni-P/SiO_2-Al_2O_3 in HDS process over a wide span of temperatures. These findings corresponding with the activation energy values for individual reactions, which combine to form the HVR hydrocracking process with the use of the studied catalyst [**Mosio-Mosiewski & Morawski, (2005)**]. It appeares that the reaction temperature and catalyst activity principally influenced the depth of HDS in the feedstock, while the effect of hydrogen pressure is limited. During hydrocracking and HDS reactions taking place at such a high excess of hydrogen, the diffusion to the catalyst within the range of pressure120-200 bar is not a conversion-limiting factor; hence, the hydrogen pressure does not control the rate of hydrogenation process [**Morawski & Mosiewski, (2006)**].

2.Combination of hydrovisbreaking and hydrocracking reaction:

By feeding heavy feedstocks to the hydrovisbraking, the overall yield of high-value products was maximized and yield of low-value coke was minimized. Therefore, combining a non-catalytic hydrovisbreaking process, followed by hydrocracking, using the sulfided catalyst WNi-P/SiO_2-Al_2O_3 was conducted at the same hydrocracking operating conditions, in order to upgrade the heavy vacuum residue, to realize the purpose of the present study. The experiments were made at a total H_2 pressure of about 120 bar and temperatures range $350°$ to $4250°C$ and constant catalyst-to-feed ratio, 0.1.

2.1.Influence of process temperatures on hydrovisbreaking/hydrocracking process:

Hydrovisreaking of the feedstock (HVR) resulted in producing an upgraded feed for hydrocracking step due to great reductions in asphaltenes (28-39%), sulfur (39-77%), which facilitate hydrocracking reactions. Therefore, the extention of hydrovisbreaking to mild hydrocracking resulted in significant changes in the yields and characteristics of distillates up to boiling range 350°C. Fig. (4 and 5) showed higher extent on conversion of feedstock to hydrocarbon gases and lighter distillates through increasing reaction temperature, i.e. from 54 wt % at 350°C to 80 wt% at 425°C, compared to hydrocracking process alone, i.e. from 53 wt% to 74 wt% at the same reaction temperatures. However, hydrogen consumption, which was indicated by pressure drop during the experiments   and amounts of gases were significantly greater when applying the combination process rather than hydrocracking. The hydrocarbon gases amount to 4-13.5 wt % of the processed feed at 350°C to 425°C reaction temperature.

The operating scheme under different reaction temperatures, induced high refining changes in quantities and qualities of gases and distillates produced (gasoline, kerosene and light gas oil). During increasing temperature, sulfur contents in the distillates was improved to be in the ranges of 0.1 to 0.01wt % for gasoline, 0.13 to 0.02 wt % for kerosene and 0.19 to 0.02 Wt % for light gas oil at 350° to 450°C, respectively. The data suggest that the success of the catalytic upgrading of the investigated feed depends on the characteristics of the employed catalyst.
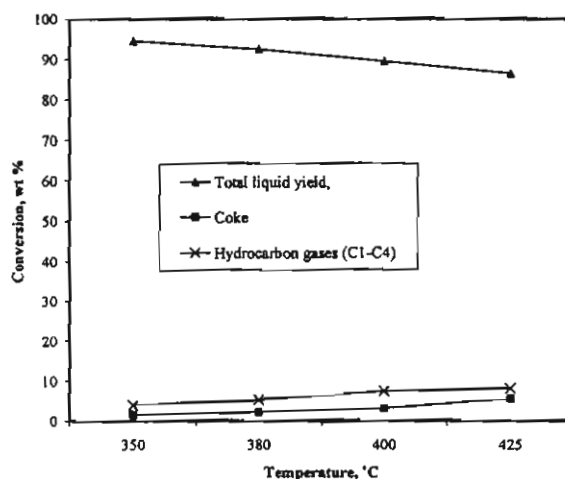

Fig. (4): Effect of process temperature on Hydrovisbreaking/ hydrocracking of HVR for total liquid yield, hydrocarbon gases and coke production.
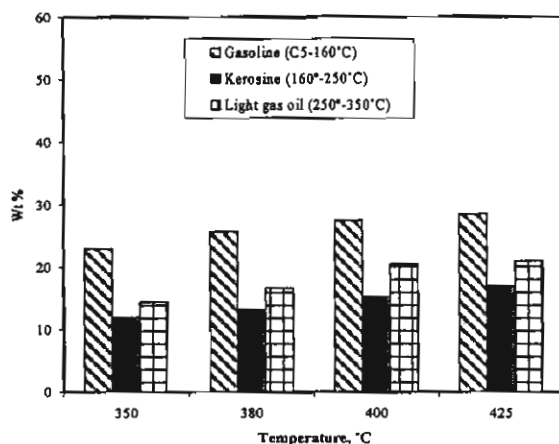
Fig. (5): Effect of process temperature on Hydrovisbreaking/ hydrocracking of HVR for distillates production

2.2.Influence of process temperatures on product composition:

The products of hydrovisbreaking/hydrocracking combination process were separated into hydrogen-rich hydrocarbon gases and total distillates. The distillates were separated into gasoline, kerosene, light gas oil (diesel) and residue.

The effects of the process parameters on the fractionated distillates from the converted HVR, under combination process, are presented in Fig. (5). When the reaction temperature increased, the heaviest residue fraction, i.e. 350°+, clearly declined. That fraction, despite the different initial boiling point as the feedstock, has a different chemical composition and somewhat different physical and chemical properties than the feed. This residue represents either boiler fuel or a heavier product used as feed for other conversion processes (catalytic cracking or delayed coking).

When the reaction temperature increased within the studied scope, the most lightweight fractions: hydrocarbon gases and gasoline grew up a little, but greater at higher temperature. The effect of temperature on the yield of gasoline was much more distinct. The yield of gasoline amounted to 23 wt % at 350°C, and then it increased to 29 wt % at 425°C. The yield of kerosene fraction reached 12 wt % at 350°C and then it increased to 17 wt % at 425°C. The same increase was observed for the light gas oil (diesel) fraction, reached 14.5 wt % at 350°C and it then increased to 21 wt % at 425°C.

The yield of residue fraction, 350°+, reached its peak value of 45.1 wt % at 350°C, and then decreasd to 20 wt % at 425°C. The reason is that heavier residue (350°C+) undergoes cracking to lighter fractions at those conditions than was produced from HVR hydrocracking. This residue can be fed to the catalytic cracking plants for the production of low sulfur fractions of motor fuels.

It is also applicable as reduced-sulfur contents fuel oil. The effect of temperature on the middle distillate fractions (160°-350°C) was much more distinct; the

yield amounted to nearly 27 wt % at 350°C and then increased to 38 wt % at 425°C. The reason is that both kerosene and light gas oil yields were increased with the temperature increase as the heavy fraction, 350°+ converted to light distillates.

2.3.Influence of process temperature on HVR hydrovisbreaking/hydro-cracking conversion:

The total conversion of the process was observed to be increased from 53.5 to 80 wt% as temperature increased from 350° to 425°C respectively. At 53.5 and 80 wt% conversion, the removal of vanadium and nickel was observed in the asphaltenes content reduction. These reductions were also reflected on the reduction in sulfur contents (HDS), the reduction in sulfur content increased with the increase of temperature, i.e. from 350° to 425°C resulted in 80 to 98 % HDS respectively. These results indicate that feeds (as the studied feedstock) with metal contents between 200 to 300 ppm (extra heavy feeds) may still be upgraded better than feeds with metal contents between 300 to 1000 ppm (problematic /or ultra heavy feeds).

The temperature effects on the total conversion of HVR, sulfur, asphaltenes and CCR were studied under the investigated operating conditions. The conversion of these constituents was higher in case of the compination process than the hydrocracking process (asphaltene conversion %, CCR residue % and HDS %).

The two-stage process might allow temperature control and a higher degree of hydrogen management. In fact, introduction of hydrogen into the reacor can have very beneficial effects. The catalytic conversion at 350°C yielded 1.4 wt % cokes. The asphaltenes content of the converted HVR was reduced, from 13 wt% in the original feedstock, to 5.2, 4.7 and 3.5 wt%, in the liquid produts, at 350°, 380° and 425°C, respectively Fig. (6). Further treatment by choice of a suitable combination process could result in a balance between process economics and products. Hence, process temperatures strongly affect the conversion of the vacuum residue as indicated in Fig. (6), and the combination scheme enhanced HVR conversion.
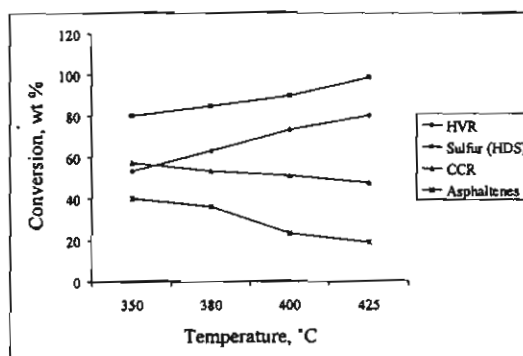

Fig. (6): Influence of process temperature on hydrovisbreaking/ hydrocracking of HVR-conversion.

3.3.The advantage of the combination of hydrovisbreaking and hydrocracking:

The obtained data indicate that hydrocracking of HVR was far more difficult than hydrocracking of crude distillates, since HVR contains considerable amounts of asphaltic and resinous compounds. The HVR, under the combination process, was operated to maximize the yield of fractions, which are lighter than the feedstock, and to subject the processed products to hydrofining under safety conditions comparing with the hydrocracking process alone. Therefore, the principle function of the catalyst employed in the present study was to eliminate the condensation reactions of highly unsaturated compounds produced during the process. Another important function of the catalyst is to promote hydrofining reactions of the hydrocracking products, inclusive principally of the hydrodesulfurization (HDS) reactions Fig. (7). HDS was a catalytic process, which was strongly dependent on the catalyst activity specification.
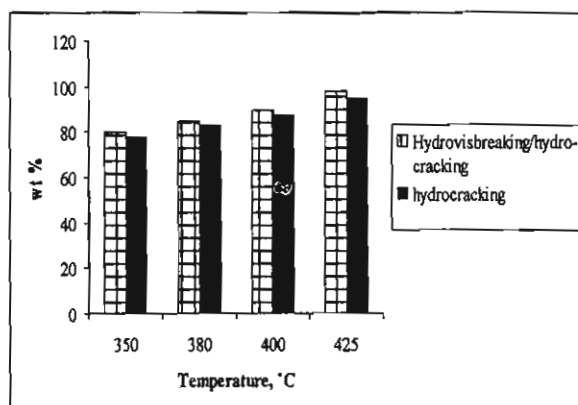

Fig. (7): Comparison of hydrodesulfurization (HDS) by hydrovisbreaking followed by hydrocracking process under variable reaction temperatures.

Running the hydrovisbreaking/hydrocracking combination process within the operating parameters, which offer mild hydrocracking of the feedstock, through the reaction temperatures 350°-425°C resulted in production of higher hydrocarbon gases between 4 and nearly 14 wt % compared with gases produced during hydrocracking reactions, and low limits of formed coke. Another advantage of applying the combination process was observed in the total process conversion of feedstock, which resulted at higher reaction temperature, above 380°C, amounted to 73.3 and 80 wt % at 400° and 425°C, respectively rather than 69 and 74 wt% at the same reaction temperatures for hydrocracking alone. This is advantageous since the basic purpose of HVR conversion, besides HDS of the feed, and its cracking products, is to yield considerable volumes of liquid fractions, which are suitable for motor fuels blending and heating fuels or can make a synthetic feed to the catalytic cracking plants to be further converted to additional volumes of motor fuels.

Finally, the pretreatment of HVR by hydrovisbreacking process before hydrocracking strongly impoved its grade, especially reductions in sulfur and asphaltenes contents, and enhanced catalytic activity of the cracking catalyst.

CONCLUSIONS

The resulted data suggest that hydrocracking reactions were not affected by mass transfer of HVR-reactant to the the active surface of the catalyst due to certain inhipition of either asphaltenes or total sulfur contents. When using the sulfided catalyst W-Ni-P/SiO$_2$-Al$_2$O$_3$, more hydrocracking and HDS activity through the combined scheme compared to hydrocracking scheme, producing lighter distillates and contaminants reduction. At higher cracking temperature, 425°C, total coke formations was nearly the same , 5%, but with 98% desulfurization and 80% conversion for the combination reactions compared with 95% desulfurization and 74% conversion for hydrocracking alone. Therefore, hydrovisbreaking process was the possible alternate option for HVR before hydrocracking process, which names a combination scheme.

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الملخص العربى

دراسة مقارنة عن عملية التكسير الهيدروجينى مع دمج لعمليتى التكسير اللزوجى والتكسير الهيدروجينى لأنتاج مقطرات خفيفة

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كلية العلوم – جامعة حلوان – حلوان – مصر

تم التكسير الهدروجينى لمقطر ثقيل ناتج الضغط المخلخل (يحتوى على:٣٠٢,٣جزء فى المليــون لمجمــوع النيكل والفانديوم و٤,٣٦% كبريت و١٣% اسفلتينات) على حفاز ذو خاصية مزدوجة مكبرت هو تنجستن- نيكل- فوسفور محمل على سيليكا والومينا، داخل مفاعل نظام التشغيلة وتحت ضغط هيدروجينى قــدرة ١٢٠ بار تحت تاثير درجات حرارة متغيرة من ٣٥٠ إلى ٤٢٥°م.

تمت مقارنة نشاط الحفاز المكبرت نحو نظام التكسير الهيدروجينى فقط مع نظام مدمج من التكـــسير الهيدروجينى للزوجة بدون إستخدام حفاز متبوعا بتفاعل التكسير الهيدروجينى باستخدام الحفاز المــشار اليــه بغرض الحصول على مقطرات خفيفة مناسبه لإستخدامها كوقود للسيارات.

و قد نتج من إستخدام الحفاز فى النظام المدمج نسبة ٨٠% تحول لمادة التغذ ية الثقيلة مع تكوين فحم بنــسبة ٥% مع نسبة عالية ٩٨% تخلص من الكبريت و كذلك ٧٣% تخلص من الأسفلتينات مقارنة بما تم الحــصول عليه في حاله استخدام نظام التكسير الهيدوجيني فقط.

لذا فان هذه النتائج تدل علي ان تطبيق التكسير الهيدروجيني للزوجه هو انسب نظام لمعالجة المقطر الثقيل قبل عمليه التكسير الهيدوجيني.