

Hydrogen Transfer Cracking of Arabian Residua in Tetralin - I

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Abstract. Heavy Arabian vacuum residua were hydrocracked non-catalytically using tetralin as a hydrogen donor solvent. A bolted closure packless high temperature/high pressure autoclave with a magdrive assembly was used batchwise for this purpose. The effects of temperature, pressure, and reactants ratio on the performance of the system were studied. The products were analyzed both qualitatively and quantitatively. Methods of analysis included: hydrocarbon group analysis using an HPLC, simulated distillation using a GC, sulfur determination and API gravity, asphaltene and carbon residue determination according to ASTM standards. Tetralin proved to be an effective donor for Arabian residua. HPLC and simulated distillation analysis showed that heavy compounds of the residuum were converted into lighter ones. When a ratio of tetralin to residuum less than 1/1 by weight was used coke formed in amounts proportional to tetralin. For ratios greater than 1/1 no coke formation was observed. A ratio of 2/1 was found to be a suitable ratio and was used in all runs. It was found that a minimum initial pressure of nitrogen gas (0.5 MPa) was sufficient to keep the reactants in the liquid state. Otherwise, pressure had no effect on the conversion. As the temperature was increased the conversion to lighter products increased. The relative percentage change of both asphaltenes and carbon residue increase with temperature. Degree of desulfurization also increased with temperature. This preliminary study was limited to use a maximum temperature of 435 °C. Increasing the temperature beyond this limit causes gas venting, and thus a loss of gaseous products. The system will be modified to work at higher temperatures by the method of rapid injection.

Introduction

The advantages of using liquid petroleum products far exceeds those of other alternative resources. Unfortunately petroleum resources are not inexhaustible, and crudes themselves are becoming heavier due to biodegradation over usage of the lighter crudes and due to evaporation losses [1]. Heavy crudes and heavy residua (the bottom of the barrel) represent a technical challenge to the petroleum industry due to the high yield of coke and the deposition of the metallic constituents. Minor volumes of the vacuum residuum are usually disposed of as road asphalt, and as a feedstock for petroleum coke and partial oxidation. The remainder is used as the major constituent of residual fuel oil [2].

The crude oil residuum is the remaining fraction after the removal of the volatile materials as a result of the nondestructive distillation (atmospheric or vacuum) of the oil. Residua fall in the category usually called heavy oils, which are defined arbitrarily but generally specified as oils low in API gravity (less than 20°) and high in viscosity and impurities particularly sulfur (usually > 2% by weight). Residua make up a significant portion of crude oils. For example residua of Arabian crudes represent more than 60% of the original crude [3]. Although reserves of residua might not last for a long time compared to those of shale oils, tar sands or coal, they can be used at least in the future, and they are cheaper and relatively easier to process.

Upgrading conversion processes [4-10] are used to convert nonvaluable materials into valuable products, such as petrochemical feedstocks, and automotive fuels. Two concepts are employed: either by carbon rejection (e.g. coking) or hydrogen addition (e.g. hydroprocessing) with the objective to increase the H/C ratio. Hydrogenation is more desirable when it can be carried out at a reasonable cost, because it produces large quantities of relatively valuable liquid products, whereas large quantities of refractory materials such as coke are produced by the carbon rejection process.

This work is concerned with a hydrogenation upgrading route involving reacting Arabian residua with a hydrogen donor solvent (tetralin), which donates atomic hydrogen to the free radicals of the residuum thus converting it into lighter products. The project is divided into stages. In the first stage residua are prepared and preliminary studies are done to test the hydrocracking reaction between tetralin and the vacuum residuum, to study the effect of temperature, pressure, and the ratio of the reactants on the performance of the system, to study the pyrolysis of tetralin, and to find out the best conditions for operation. In the second stage comparison between different cuts is performed, the kinetics of the hydrogenation reactions are studied, and simple models are built and tested. In this paper the results of the first stage are presented and discussed.

Tetralin: A Hydrogen Donor Solvent

Hydrogen donor materials have the ability to release hydrogen and thereby to convert heavy hydrocarbons to more valuable lower boiling products. Paraffin and single-ring naphthenic compounds are ineffective hydrogen transfer agents; a condensed ring naphthenic compound such as decalin is somewhat effective, whereas a mixed naphthenic-aromatic condensed-ring compound such as tetralin is much more effective.

Tetralin is the trade name for 1,2,3,4-Tetrahydronaphthalene ($C_{10}H_{12}$) with a molecular weight of 132.2, a boiling point of 207°C, a flash point of 75°C, and a specific gravity of 0.967 at 20°C. It has been used as a hydrogen donor solvent in heavy oils and residuum upgrading, in coal liquefaction, and in wood liquefaction [11-13].

Greensfelder [14] treated crude residuum noncatalytically with tetralin and hydrogen in the first stage, followed by catalytic hydrogenation. These processes do not

prevent contamination of the catalyst by the metals and resins in the residuum. Early investigation did not appreciate the ability of condensed ring naphthalene-aromatics to donate large amounts of hydrogen readily in a manner which prevents coke formation.

The use of tetralin as a hydrogen donor during thermal cracking of various residua, asphalts, and lubricating oil extract was investigated by Carlson *et al.* [12] to determine its general utility. Conversion ranged from 50 to 94% with very little coke and low dry gas yields. Feed stock examined included: Kuwaiti asphalt, Hawkins asphalt, McMurry tar, Elk Basin residuum, Coleville residuum, Bachaquero residuum and lubricating oil extract.

Yoshida *et al.* [15] subjected the atmospheric residuum from Khafji crude to thermal cracking at 420-460°C in the presence of a diluent, namely, nitrogen, hydrogen, or tetralin as a hydrogen donor. The Khafji topped crude was markedly susceptible to thermal cracking. Significant changes in physicochemical properties of the visbroken residua were observed, namely a decrease in viscosity and an increase in Conradson carbon. Bakshi and Lutz [16] discussed the advantages of adding a hydrogen donor to an existing visbreaking process. The process gives a substantially higher conversion of residual fuel oil to gas oil and higher products than does conventional visbreaking.

Studies on the pyrolysis of tetralin showed that the following processes might occur: disproportionation, dehydrogenation to naphthalene, and cracking. Extensive decomposition is observed only beyond 500°C. At 400°C ethylbenzene, propylbenzene, toluene and indane are formed. The first three may be regarded as products of the side chain cracking of butylbenzene which itself is formed from the α -ring opening of tetralin [17-20]. Gaseous products included hydrogen, methane, ethane, propane, propene and butane, with different proportions.

Experimental Set-up and Procedure

The reactor is a bolted closure packless autoclave with a magne-drive assembly. The bolted closure consists of the body, cover, closure gasket and cap screws. The gasket is made of stainless steel to withstand the high temperature range of 400-470°C. Accessories of the bolted closure consisted of the following: safety head assembly, pressure gauge, sample tube and valve assembly, cooling coil, thermowell, charging opening, agitator, digital tachometer indicator, and furnace and temperature control system.

Figure 1 shows the arrangement of the experimental set-up including control boxes and the cooling water circulation. The high pressure/high temperature reactor was connected to a nitrogen cylinder through a high pressure regulator. This arrangement enables the reactor to be pressure tested and allows also the reaction to be run under nitrogen atmosphere. Cold water from a cooling bath can be circulated in parallel through the cooling coil of the reactor as well as through the shaft of the magnetic drive. The latter is always practiced when the stirrer is on.

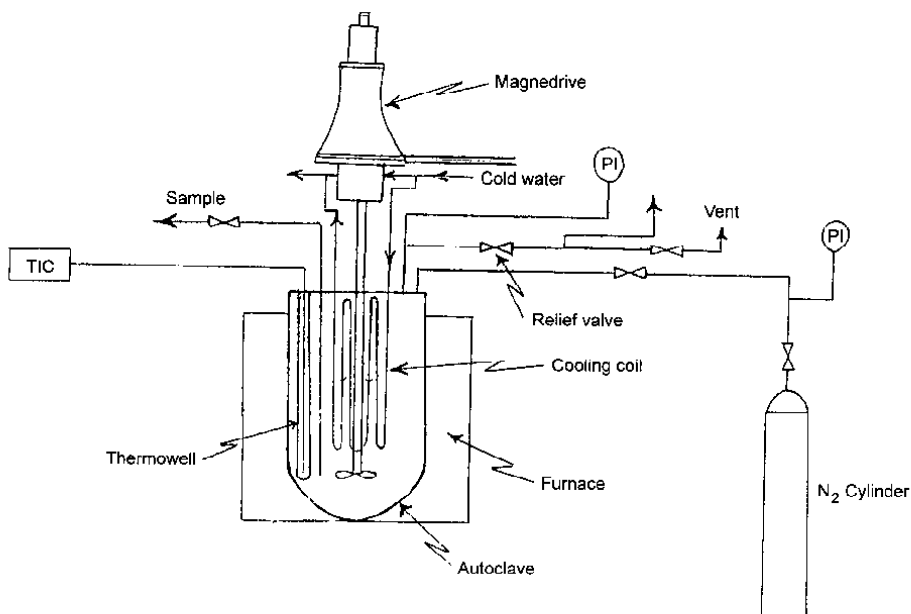


Fig. 1. Experimental setup.

Trial-and-error was followed in tuning the digital controller. The proportional band was set at 6% and the integral time at its maximum (199 min), while the derivative time was set at zero value. This situation created an offset. Integral time was decreased until this offset was eliminated. However, this situation created oscillations and the derivative time was altered until cycling was eliminated. A sample of residuum was heated at 400°C. The best setting for this case is as follows: proportional band 6%, integral time 10 min, and derivative time 5 sec. It has been found that settings are function of the material used. Thus for a mixture of tetralin and residuum settings differ. This is also the case with different temperatures. Occasionally, cooling was used to reset the temperature in certain cases during operation.

Preparation of the residua

Arab heavy crude oil was obtained from the Arabian Oil Company (ARAMCO). The heavy Arabian crude oil was first distilled at atmospheric conditions followed by vacuum distillation at 10-12 mmHg using a tilting-funnel distillation column assembly. This yielded a residuum of 343°C+ which is then further distilled under high vacuum in a wiped wall molecular still, and this operation yielded a vacuum residuum of 565°C+. Another residuum was obtained by atmospheric distillation of the crude followed by vacuum distillation using a simple distillation column. This operation yielded a residuum of 350°C+.

Experimental procedures

Tetralin and the residuum, in a precise ratio, were mixed well to obtain a homogenous mixture. A sample of 150 g was loaded into the autoclave, which was purged by nitrogen to remove air as much as possible. After that it was pressurized by nitrogen to 60 bar to test for leakage. The nitrogen pressure was brought down to the initial operating pressure (mostly 5 bar).

The system was heated under proper controller settings to take the system down to the set point temperature. Cooling water was circulated to the agitation shaft, which was running at a speed of 608 rpm to obtain uniform temperature distribution.

The reaction was permitted to proceed isothermally for 90 min. started from the time it reached the set point. Runs were repeated for different temperatures and different initial pressures. At the end of the experiment the reactor was left to cool at ambient conditions, occasionally using cooling water.

The cooling temperature and pressure were recorded and the products were collected. Liquid products were collected through the sample line in a preweighed beaker, weighed and then kept for analysis. Gases were collected in a rubber balloon after being evacuated under a vacuum line for about 15 minutes, and then sent for analysis. After that the autoclave was cleaned thoroughly using toluene or benzene or a mixture of them. The washing liquid was sucked out by vacuum.

Pyrolysis of tetralin

Tetralin was subjected to the same pyrolytic conditions in order to see what changes would occur. The autoclave was cleaned thoroughly, and several times by toluene, or benzene, or a mixture of them in order to remove any dirt or oil from previous experiments. The reactor was flushed with these solvents after being closed and pressurized by nitrogen at 0.5 MPa, in order to clean sample lines and gas outlets. Then, after being disassembled, the reactor was cleaned by tetralin alone. In all runs liquid was sucked out through a vacuum line.

A sample of 100 g of tetralin was loaded in the autoclave which was then pressurized at 0.5 MPa of nitrogen for leakage test. The reactor was flushed several times by nitrogen to remove air. The pressure of nitrogen was set at 0.5 MPa. The reactor was heated to the set point at 425°C, then left for 90 min. after reaching the set point.

At the end of the experiment, the reactor was allowed to cool. The final pressure and the pressure after cooling both were recorded. Gases were collected in a rubber balloon after it had been evacuated for 15 min. by a vacuum line to eliminate air as much as possible.

Analytical Techniques

Thermal hydrogenation results in different gaseous and liquid products. In presence of excess tetralin, no coke is formed. Liquid products have been analyzed by different techniques [21]:

1. Total sulfur by ASTM D1551 method.
2. API gravity by ASTM D941.
3. Qualitative analysis of the hydrocarbon groups using an HPLC.
4. Asphaltenes by ASTM D2007.
5. Simulated distillation by 5800A gas chromatograph corresponding to ASTM D2887-73 methods.
6. Carbon residue by Ramsbottom method corresponding to ASTM D524.

Gaseous products were analyzed by Refinery Gas Analyzer 5880 A.

Results and Discussion

Characterization of the residua

Experiments of this stage were done on a fraction boiling above 350°C. Other fractions and commercial cuts will be used for comparison in another stage. Table 1 summarizes some characteristic properties of this fraction.

Table 1. Properties of the residuum

Property	API	Sulfur wt%	Asphaltene wt%	Carbon residue wt%
Value	3.0	5.8	33.3	26.0
ASTM method	D941	D1551	D2007	D524

Definition of residua and other heavy fractions is arbitrary, hence for purposes of comparison the best way may be to specify the cut range and the conditions used to obtain the residuum. The cut range of the residuum was determined by the method of simulated distillation, which is a gas chromatographic technique by which the percentage distilled of the fraction is plotted against the true boiling range.

Preliminary studies

Hydrogenation by a donor solvent can be described by the mechanism of the free radicals [22,23]. However, free radicals are not expected to be formed at temperatures less than 400°C which is close to the initial boiling point of the residuum. Also the relative percentage change of sulfur content was not significant at 400°C, so it was decided to increase the temperature. It was found that operating at temperatures higher than 435°C with pressures higher than 0.5 MPa of nitrogen created pressures greater than the set point. These restrictions limited the work to 435°C only. Pressure of nitrogen is needed to keep reactants in the liquid form. A series of runs were done to set the proper operating conditions of temperature and pressure.

Initially any change due to the reaction between tetralin and the residuum was observed in terms of the change in the chromatographic peaks. A sample of the feed (tetralin mixed with the residuum at a certain ratio) and a sample of the product were subjected to analysis by HPLC and hydrocarbon groups, and the results compared in a qualitative manner. Followings are some of the observations:

- New peaks appeared for samples of product of the residuum at 400°C. (Figs. 2 and 3).
- The height of these peaks increased for temperatures of 425 and 435°C. (Figs. 4 and 5).
- Samples of product at 400°C and different pressures (1.5 and 2.5 MPa) showed that pressure had no effect on the product distribution when it is beyond 0.5 MPa. (Figs. 6 and 7).
- Hydrocarbon group analysis detected the appearance of new peaks in the saturates and the aromatic domain. (Figs. 8 and 9).

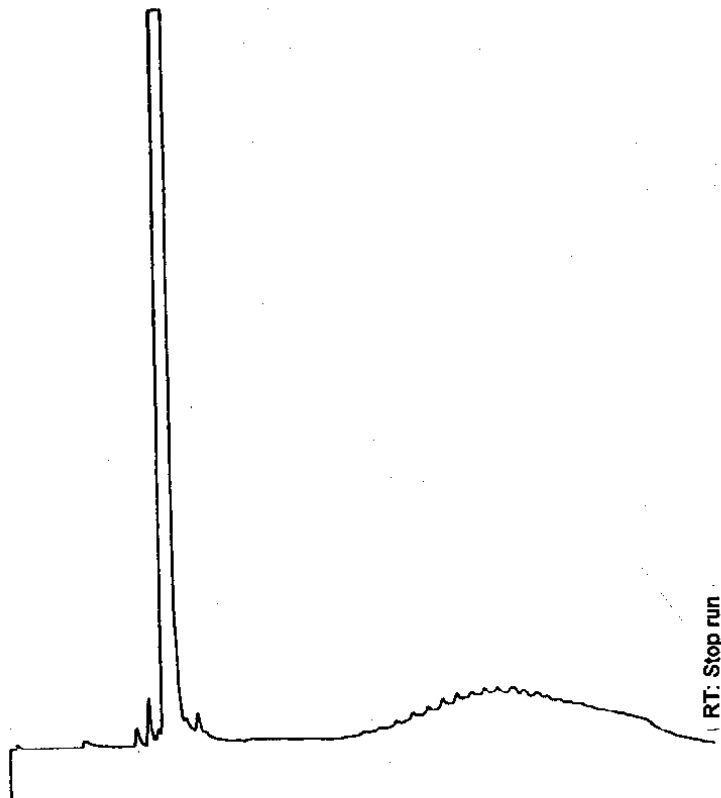


Fig. 2. GC chromatogram of the residuum in tetralin.

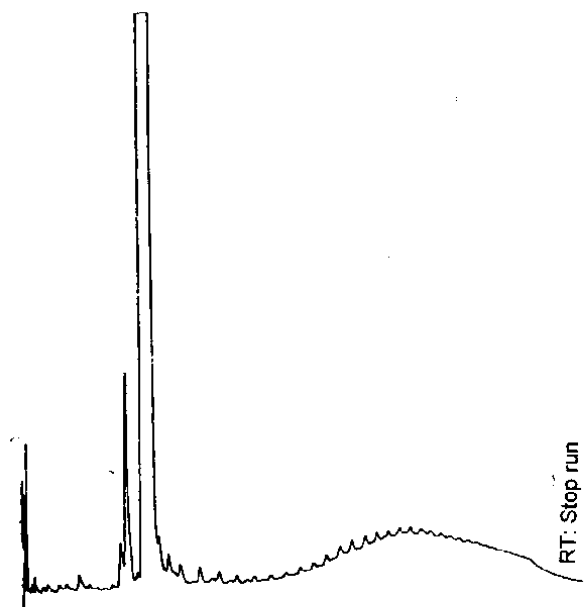


Fig. 3. GC chromatogram of the products of the residuum in tetralin at 400°C.

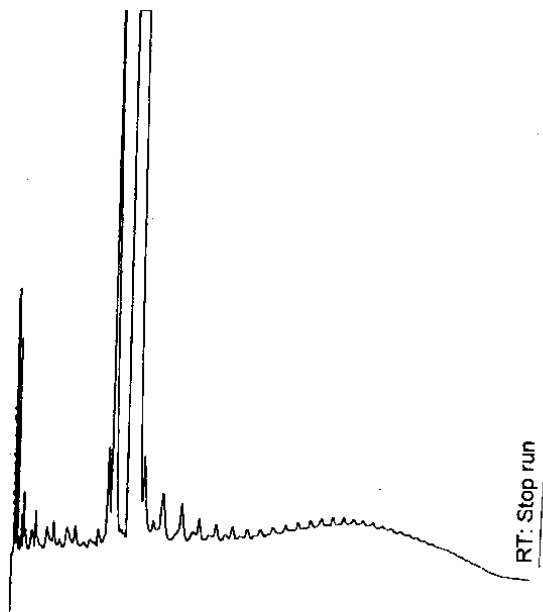


Fig. 4. GC chromatogram of the products of the residuum in tetralin at 425°C.

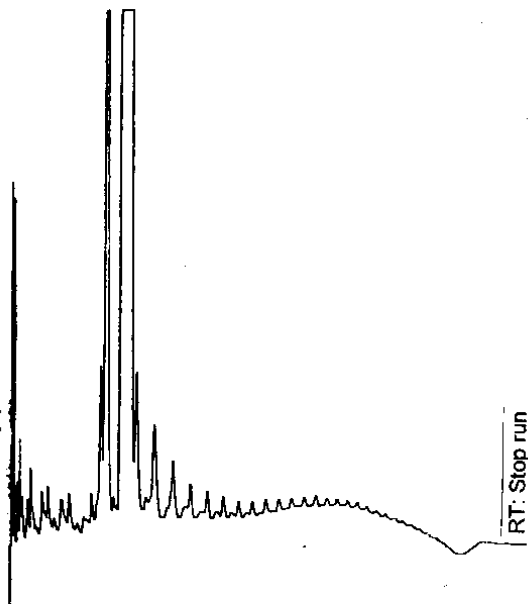


Fig. 5. GC chromatogram of the products of the residuum in tetralin at 435°C.

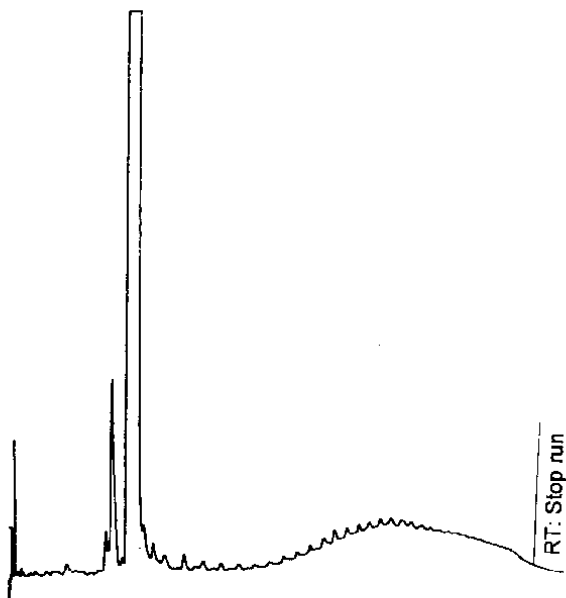


Fig. 6. GC chromatogram of the products of the residuum in tetralin at 400°C and 1.5 Mpa.

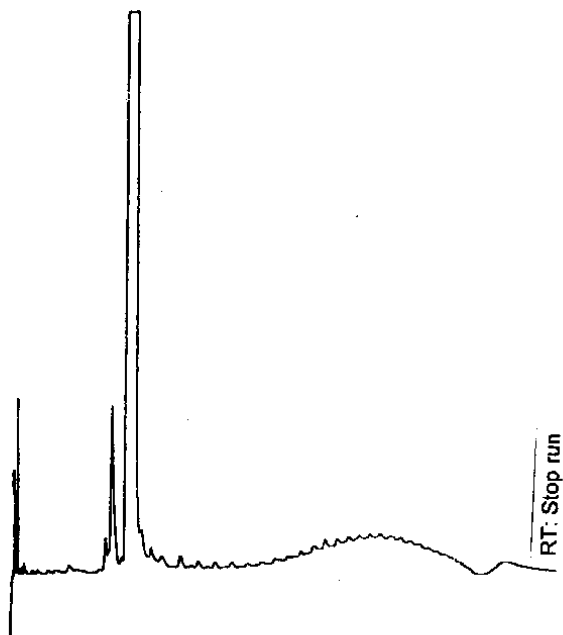


Fig. 7. GC chromatogram of the products of the residuum in tetralin at 400°C and 2.5 Mpa.

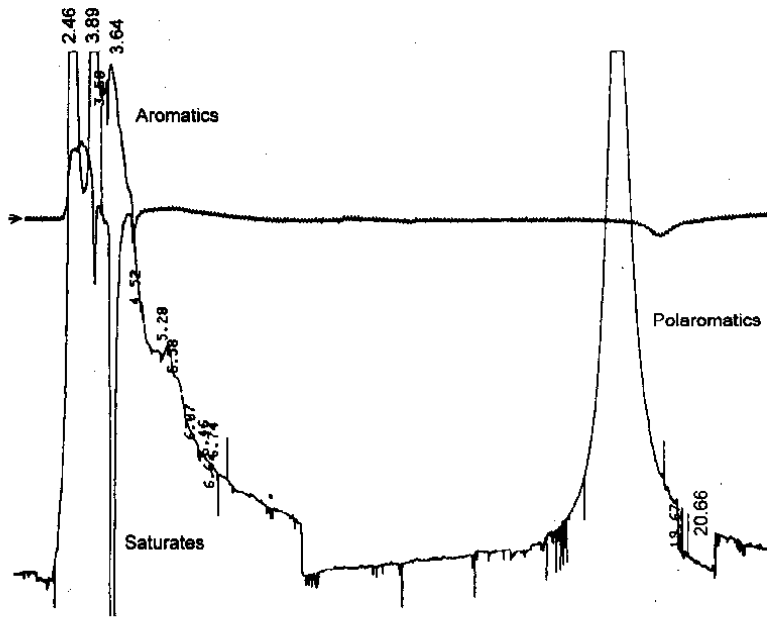


Fig. 8. Hydrocarbon group analysis of the residuum in tetralin.

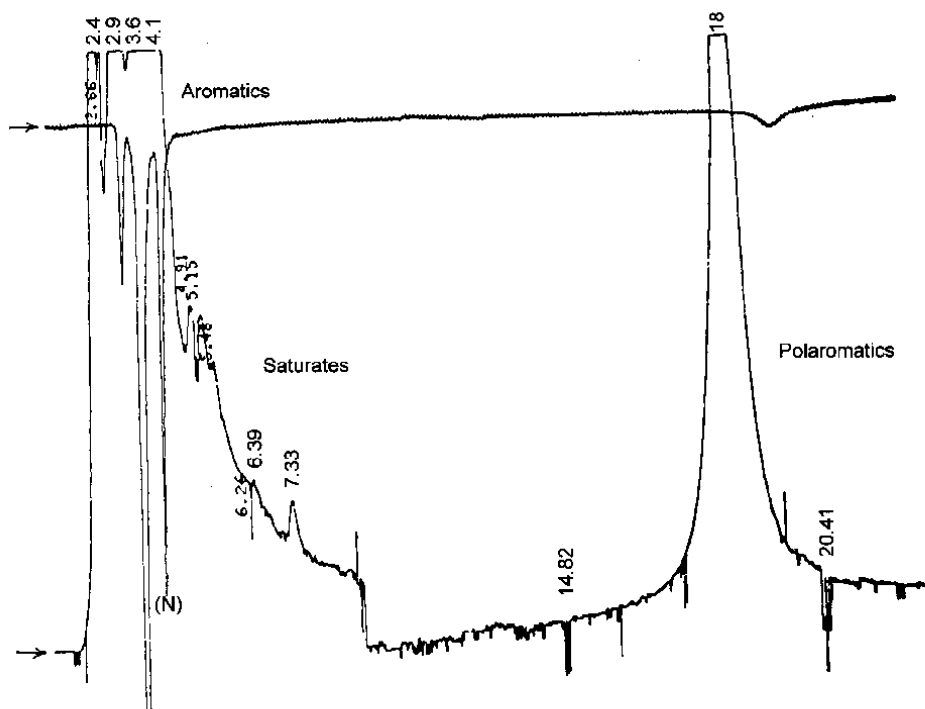


Fig. 9. Hydrocarbon group analysis of the products of the residuum intetralin at 425°C.

It is clear from the above observations that many new peaks appeared, which means that heavy compounds (asphaltenes and resins) have been converted into higher ones, i.e., they have been hydrocracked by tetralin.

Reactants ratio

Ratio of tetralin to residuum is an important factor in thermal hydrogenation because it determines which reactant should be in excess, and whether coke is formed. Samples of the residuum, mixed with tetralin at 400°C and 4.0 MPa of nitrogen pressure were reacted for 90 min., and products were analyzed for coke formation. Results showed that traces of coke formed when the ratio of tetralin to residuum was 1/1, and that the amount of coke increased for ratios of 1/2 and 1/5. With a ratio 2/1 no coke formed. In fact this ratio was chosen for all runs, because it inhibited coke formation and guaranteed an excess of tetralin for hydrogen consumption.

Pressure effect

Pressure is needed to keep reactants in the liquid form, thus preventing the solvent from evaporation. It has been found that an initial pressure of 0.5 MPa is sufficient for this purpose. This can be seen from Table 2. There is no effect of pressures higher than 0.5 MPa on the API gravity, sulfur content, or asphaltene content. It was not possible to

try higher values of pressure without exceeding the pressure set point (10 MPa) and thus losing gaseous products. All runs were performed at 400°C. It was difficult to run at higher temperatures and at pressures higher than 1 MPa for the mentioned reasons.

Table 2. Pressure effect

Pressure, bar	API	sulfur, wt%	Asphaltene, wt%
5	13.5	1.45	4.9
15	13.4	1.46	4.9
25	13.4	1.46	4.9
ASTM method	D941	D1551	D2007

Residuum cut: 350°C¹

Temperature: 400°C

Reaction time: 90 min.

Ratio of tetralin to residuum: 2/1 (wt/wt)

Temperature effect

The use of a high temperature is essential in order to release free radicals. This also increases the free energy and the rate of reaction. Conversion was studied at three different temperatures, namely: 400, 425, and 435°C, in terms of asphaltene content of the products and their carbon residue. Results are presented in Table 3, which show that the change in the content of asphaltene and carbon residue increased as temperature increased. Increasing the temperature beyond this level caused gas venting and a loss of gaseous products. The system will need to be modified in the future in order to work at higher temperatures.

Table 3. Carbon residue and asphaltene content of the products at different temperatures

Product temperature, °C	Carbon residue, wt%	Asphaltene content, wt%
400	4.3	4.9
425	3.7	3.5
435	3.3	2.5
ASTM method	D524	D2007

Initial pressure: 5 bar

Reaction time: 90 min

Tetralin to residuum ratio: 2/1

Refinery gas analysis showed that the main gaseous products are: hydrogen, carbon dioxide, propane, propylene, i-butane, n-butane, hydrogen sulfide, ethylene, ethane and methane. Gaseous products amount approximately to 2 wt%. Hydrogen amounts to about 35% by volume, methane 28% and hydrogen sulfide 5%. These were the gaseous products at 425°C. Hydrogen could be a hydrocracking product or a dehydrogenation product of tetralin. Oxygen which is not a gaseous product probably entered during sampling process. Carbon dioxide might be a product of indirect combustion especially with low olefins.

Pyrolysis of tetralin

Tetralin was heated to 425°C for 90 min under an initial pressure of 0.5 MPa. An extra pressure of 0.25 MPa of gases was observed upon cooling. The gaseous product was analyzed by a refinery gas analyzer. Hydrogen, oxygen, nitrogen and methane were observed. Methane could be a product of disproportionation of tetralin to methylindane and subsequently to indane, a reaction which has been detected in the range of 400-500 °C. Oxygen probably came with nitrogen or air during sampling. Gas produced totals approximately to 0.1% by weight of the total sample.

The liquid portion was analyzed by HPLC. Results indicated the presence of aromatics and saturates. For gross identification and comparison, a sample of tetralin alone, a sample of naphthalene alone and a sample of an equal mixture of them were injected into the HPLC. Peaks of aromatics and naphthalene seem to be due to impurities. Insignificant dehydrogenation of tetralin to molecular hydrogen could have happened. Tetralin is not liable to crack under the conditions considered in this study.

Conclusion

- ◆ A ratio > 1/1 (wt/wt) of tetralin to residuum inhibits coke formation and provides an excess of tetralin. Ratios less than this produce coke in amounts proportional to the ratio. A ratio of 2/1 was found to be suitable and was used in all runs.
- ◆ As the temperature increases the conversion to lighter products increases. About 50% change of the asphaltenes is observed at 425°C.
- ◆ Degree of desulfurization increases with temperature.
- ◆ An initial pressure of nitrogen of 0.5 MPa is sufficient to keep reactants in the liquid state. Otherwise, the pressure has no significant effect.
- ◆ Tetralin can be considered as an effective hydrogen donor for Arabian residua.
- ◆ Tetralin is not liable to cracking under the conditions of this study.

Recommendations

This preliminary study revealed several important points that must be considered in a continuation of this work:

- It is recommended that, in future work, products be separated into fractions, and each fraction be analyzed separately. Comparison on this basis is better and more obvious.
- When separated the spent donor can be rehydrogenated, which means that the solvent will be used at the initial stage only.
- It is believed that conversion will increase if cycling is used, i.e., after separation into fractions, the heavier one to be treated again until a considerable conversion is obtained.
- Other donors may be tried provided they are of the fused naphthenic-aromatic type. Also petroleum fractions such as light cycle oil may be used.

Separation into fractions enables one to study lumped kinetics in terms of certain variables, e.g., sulfur or asphaltene. However, the system need to be modified for rapid injection. Model compounds can be used to study hydrodesulfurization with donor solvents.

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التكسير الهيدروجيني لبقايا النفط العربي باستخدام التترالين

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(قدم للنشر في ١٤/٠٥/٢٠٠٠م، وقبل للنشر في ٠٨/٠١/٢٠٠١م)

ملخص البحث. أجريت في هذا البحث دراسة مبدئية لتكسير بقايا النفط العربي الثقيل عن طريق الهدرجة باستخدام وسط مانح للهيدروجينات هو التترالين، دون استخدام الحوافز، حيث جرى ذلك في مفاعل الأوتوكلاف والذي يعمل بالتشغيل المتقطع تحت ضغط عال وحرارة مرتفعة. تضمن البحث دراسة تأثير كل من نسب المواد المتفاعلة، ودرجة الحرارة، والضغط، وقد جرى تحليل النتائج كما وكيفا باستخدام طرق تحليلية عديدة هي: طريقة تحليل المجموعات الهيدروكربونية، وطريقة التقطير المحاكي، وطريقة تعيين المحتوى الكبريتي، وطريقة إيجاد الوزن النوعي النفطي، وطرق تعيين المحتوى الأسفلتي والبقية المتضخمة وفقا لمعايير ASTM.

أثبت هذا البحث مبدئياً أن التترالين مانح جيد وملائم لبقايا النفط العربي، حيث تبين بالتحليل الكيفي تحول المكونات الثقيلة في البقايا إلى مكونات خفيفة بفعل الهدرجة. أما من حيث العوامل المؤثرة فقد لوحظ بالنسبة للمواد المتفاعلة تكون الفحم الكربوني عندما كانت النسبة المستخدمة بين التترالين والبقية النفطية ١/١، ولم يتكون الفحم عندما كانت النسبة أعلى من ١/١، ووجد أن النسبة ١/٢ ملائمة للعمل بها، لذا فقد استخدمت في كل التجارب. أما من حيث تأثير درجة الحرارة والضغط فقد تبين من هذا البحث أن حداً أدنى من الضغط الغازي لا بد من توافره من أجل الحفاظ على المواد المتفاعلة في حالة السيولة (حيث يكفي ضغط من النيتروجين قدره ٠.٥ ميغاباسكال في بداية التشغيل)، وليس للضغط أي تأثير آخر على التحول الكيماوي. كما تبين أن التحول يزداد بارتفاع درجة الحرارة، حيث لوحظ ارتفاع نسبة تحول كل من الاسفلتين والبقية المتضخمة مع زيادة درجة الحرارة. كما لوحظ أن درجة تحويل الكبريت المنزوع ازدادت بارتفاع درجة الحرارة. وقد جرى العمل في المدى ٤٠٠ - ٤٣٥°م حيث إن أي زيادة خارج هذا المدى تؤدي إلى تجاوز حد الأمان في النظام وهروب الغازات الناتجة، وهذا يعني ضرورة تعديل النظام من أجل تعمق الدراسة وتوسيعها مستقبلاً للتمكن من دراسة حركية التفاعلات الناتجة.