Properties and Composition of Products from Hydrotreating of Straight-Run Gas Oil and Its Mixtures with Light Cycle Oil Over Sulfidic Ni-Mo/Al₂O₃ Catalyst

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1. INTRODUCTION

The consumption of motor fuels is increasingly rising. That is why it is necessary to process less favored fractions for its production.

The production of motor fuels includes several refinery processes. In terms of the amount of processed material, fluid catalytic cracking (FCC) belongs to one of the most important processes. Products resulting from FCC are distilled into gases, cracked naphtha, a middle distillate known as light cycle oil (LCO), and a distillation residue known as heavy fuel oil or slurry oil. It is an effort to use most of the fractions from the FCC to produce motor fuels. Due to the high sulfur content, naphtha and the middle distillates are usually desulfurized. Hydrotreating is the most used process for the desulfurization of medium and heavy naphtha and light cycle oil. Cracked naphtha has a relatively high octane number, after desulfurization, and it is usually used as a component of gasoline.

Light cycle oil has a distillation range typically between 180 and 340 °C. It is usually used for the production of diesel fuels and sometimes for the production of various kinds of fuel oils. A promising option for upgrading the LCO is its hydrotreatment followed by hydrocracking to produce high-value benzene, toluene, and xylene (BTX)-rich aromatics. The sulfur and nitrogen contents in the LCO depend mainly on the contents of these elements in the raw material processed by the FCC. A significant part of the sulfur contained in the LCO is bound in alkyl dibenzothiophenes whose desulfurization is difficult. Nitrogen is especially bound in alkyl carbazoles, which further hinders the LCO hydrotreatment. Another problem in the processing of the LCO to the component for diesel fuels is its high content of aromatics, which causes its high density and low cetane number.

Diesel fuels must meet the requirements of the quality set by the standard valid for each country. In the European Union, diesel must meet strict legislative criteria, such as a very low sulfur content (max 10 mg·kg⁻¹) and a low content of
polycyclic aromatic hydrocarbons (max 8 wt %). Due to its properties, a hydrotreated LCO may be applied in the production of diesel fuels in a limited amount.9

LCO is usually refined together with other middle distillates, which are commonly used for the production of diesel fuels. During hydrotreating, the sulfur, as well as other heteroatoms, is removed by hydrogenolysis. Furthermore, the desired hydrogenation of polycyclic aromatics to monoaromatic compounds and, to a small extent, the hydrogenation of monoaromatic compounds to saturated hydrocarbons is achieved. This reduces the density and improves the cetane number of the obtained product. The cracking of LCO is rather undesirable because it would yield less product to be used in diesel production.10

A CoMo/Al2O3 or NiMo/Al2O3 catalyst, a temperature range of 340–400 °C, and a pressure of 4–8 MPa are the most frequently used for the hydrotreating of middle distillates. A product with better properties is usually obtained at higher reaction temperatures, higher pressure, or lower space velocity of the feedstock.11,12

The nature of the support generally plays an important role in the catalytic activity of the prepared catalysts.13 Al2O3 is the most widely used support of hydrodesulfurization (HDS) catalysts because it is highly stable, it has a relatively large surface and porosity, and is relatively inexpensive.14 Conventional alumina and most of the other supports are not inert supports under the hydrotreating reaction conditions. They promote isomerization reactions depending on their acidic character. The dispersion, average stacking, and length of the MoS2 or WS2 slabs and the way in which the metal sulfides are bonded to the support surface are influenced by the support. The support could allow the migration of the active promoters, such as Co or Ni, into its more external surface, forming a more active catalyst.13,15

The use of catalysts with carbonaceous materials as a support shows significant activity for HDS reactions on a laboratory level. The availability of the methods for the preparation of oxides, such as ZrO2, TiO2, and MgO, which have a large surface area, generates greater interest in these materials as components of bifunctional HDS catalysts.13 To use their high activity, their mixtures such as TiO2−Al2O3 and ZrO2−TiO2 are used in many cases.13,16,17

Slightly acidic nonpolar supports (such as TiO2 and powdered activated carbon) appear to be better components of bifunctional catalysts used for the HDS of aromatic-rich feedstock, such as LCO. The HDS activity of the TiO2-based catalyst was better than that of the Al2O3 one and the cracking was, on the contrary, lower.6

NiMo catalysts on the SiO2−Al2O3 support, which is more acidic than Al2O3, can also be used for the LCO processing of diesel fuels. Under the standard hydrotreating conditions, the catalyst exhibited predominantly desirable reactions of hydrogenation, skeletal isomerization, and the removal of polyaromatics, which were mainly converted to monoaromatics. Hydrocracking reactions, which are not too desirable during hydrotreating, were also observed, but part of the product of these reactions can be used as a component in the gasoline pool to increase the octane number of the gasoline.18

The addition of LCO to the straight-run gas oil (SRGO) predominantly increases the density and sulfur, nitrogen, and aromatic contents of the prepared mixture. On the contrary, the viscosity and cetane index (CI) of the mixture decrease. The properties of the mixture have an effect on the hydrotreatment itself and, of course, on the properties of the final products. The more the LCO added to the raw material, the lower the quality of the final products gained, mainly due to the higher aromatic hydrocarbon content. The more severe reaction conditions (higher temperature or pressure or lower space velocity) must be used for hydrotreating the raw material with a high LCO content compared to the raw material with a low LCO content to obtain products meeting the EN 590 characteristics.10,19

Escalona et al.18 observed that the monoaromatic and polycyclic hydrocarbons increases and the paraffin yield decreased at a higher temperature (>360 °C), and the naphthen (cycloalkanes) yield decreased after 380 °C. It is because the hydrogen-transfer reaction and naphthene dehydrogenation, being equilibrium-controlled, is more favored at a higher temperature.18 A decrease in the monoaromatic content and an increase in the polycyclic content in the LCO hydrotreating products with a rising reaction temperature in the range of 340–380 °C were also announced by Dagonikou et al.1 However, when they hydrotreated LCO with a lower end of distillation, there was no increase in the polycyclic content with increasing reaction temperature.

Danaher et al.20 found that under mild conditions of LCO hydrotreating, the diaromatics and polyaromatics were mainly converted to monoaromatics. At higher severities, the amounts of diaromatics and polycyclics continued to reduce because of their conversion into monoaromatics. The amount of formed monoaromatics increased to a certain maximum depending on the increasing sharpness of the reaction conditions and then decreased as they were converted into saturated compounds. The increase in the amount of aromatics was attributed almost totally to the production of naphthenic hydrocarbons.20 An increase in the polycyclic content was not observed under severe reaction conditions because a decrease in product density was used to determine the severity of reaction conditions. A pressure of 20 MPa was used under the most severe reaction conditions, which shifted the equilibrium of aromatic ring hydrogenation toward cycloalkane rings.20 If the sharpness of the reaction conditions is expressed by increasing temperature, the content of diaromatics and polycyclics in the hydrotreating products increases from a certain temperature depending on the increasing temperature (sharpness of the reaction conditions).18

Higher quality LCO can be obtained by reduction of its initial boiling point at the expense of the heavy naphtha. Not only more LCO is obtained, but it also reduces its density as well as the sulfur, nitrogen, and polycyclic hydrocarbon contents, which lead to its easier hydrotreatment.9 The second option is to reduce the final boiling point of the LCO, which also leads to the improvement of its properties, but it reduces the LCO yield.5,19

Increasing the amount of LCO in the feedstock for hydrotreatment can also be done by the addition of vegetable oils to this feedstock.21 The product from vegetable oil hydrotreatment contains mainly C15−C18 alkanes that have a low density; therefore, more LCO with a high density can be used in the raw material blend.

Another possibility of increasing the amount of LCO in diesel fuels is the subsequent catalytic hydroconversion of the desulfurized and dearomatized LCO over Ir/Y zeolite or Pt/Y zeolite catalysts.22
Another fraction from the FCC usable for the production of diesel fuel is desulfurized heavy naphtha having a distillation range of usually between 150 and 200 °C. The tendency to use heavy naphtha for the production of diesel fuel increases as the consumption of gasoline in Europe decreases and the consumption of diesel fuel increases.23

FCC naphtha probably can shift the sulfur distribution in the raw material toward more reactive compounds along with a bit lower total sulfur content for the final raw material mixture. Additionally, the FCC naphtha most likely enables a partial shift of the hydrocarbon material and reactive sulfur compounds to a gas phase, which results in higher reaction rates of the reactions taking place on the catalyst surface.2,24

This study aimed at determining the effect of LCO addition to the straight-run gas oil (SRGO) on the properties and composition of the products from their hydrotreatment. The hydrotreatment was carried out using a sulfided NiMo/Al2O3 catalyst, which has a higher hydrogenation activity than the commonly used CoMo/Al2O3 catalyst. The effects of the addition of the desulfurized kerosene from the crude oil distillation and desulfurized heavy naphtha from the FCC on the properties of the prepared products were also measured.

2. RESULTS AND DISCUSSION

2.1. Feedstock Characterization. Two straight-run gas oils (SRGO-S and SRGO-W) and two light cycle oils (LCO-S and LCO-W) from FCC were used for preparing the raw materials for hydrotreatment. The raw materials marked with “S” were used in the Královo refinery (Unipetrol RPA) to produce a “summer” diesel fuel, and the feedstocks marked with “W” were used to produce a “winter” diesel fuel. The hydrotreating parameters were set to obtain a product with the sulfur content of about 10 mg·kg⁻¹ from the summer blend prepared from 10 wt % LCO-S and 90 wt % SRGO-S and from 10 wt % LCO-W and 90 wt % SRGO-W.

Table 1. LCO Quantity in the Raw Material and the Designation of the Feedstocks and the Obtained Hydrotreatment Products

| LCO quantity in the feedstock [wt %] | feedstock designation product designation | summer blend | winter blend | summer blend | winter blend |
|--------------------------------------|------------------------------------------|--------------|--------------|--------------|--------------|
| 0                                    | SF0                                      | WF0          | SH0          | WH0          |
| 5                                    | SF5                                      | WF5          | SH5          | WH5          |
| 10                                   | SF10                                     | WF10         | SH10         | WH10         |
| 15                                   | SF15                                     | WF15         | SH15         | WH15         |
| 20                                   | SF20                                     | WF20         | SH20         | WH20         |

The properties of SRGO-S and SRGO-W did not differ much (Table 2); the LCO-W had a slightly lower temperature at which 95 vol % sample was recovered (T95). The LCO-W had higher contents of sulfur and nitrogen than the LCO-S. Also, it had a higher content of polycyclic aromatic hydrocarbons (PAHs) and a higher T95, which caused its higher density and viscosity (Table 2).

Both LCOs had much higher contents of sulfur, nitrogen, and PAHs compared to the SRGOs. The high content of PAHs in the LCOs caused their high density and low cetane index (CI). The cold filter plugging point (CFPP) of both LCOs was much better than the ones of the SRGOs.

The amount of distillate recovered at 250 °C (R250) was higher and the temperature at which 95 vol % sample was recovered (T95) was lower for the LCO-S compared with the LCO-W. The T95 of the LCOs was at a lower temperature than the SRGO ones (Table 2).

The yields of the liquid products from the hydrotreating of the summer blends were between 98.5 and 99.2 wt %, and the product yields from the hydrotreating of the winter blends were between 97.8 and 98.7 wt %. The high yields indicate minimal cracking during hydrotreating. The slightly lower yields of the products from the winter blends were caused by a slightly higher content of hydrocarbons with a lower boiling point in SRGO-W compared to SRGO-S.

The yields of the distillation residue from the stabilization of the product from the hydrotreating of the summer and winter blends were about 99.5 and 98.5 wt %, respectively. The yields of products boiling at 150 °C were about 0.35 and 1.3 wt % from the summer and winter blends, respectively.

2.2. Hydrotreating Product Characterization. The properties of the real feedstocks and stabilized products from their hydrotreatment are presented in Tables 3 and 4. The increasing LCO content in the feedstock led to an increase in the contents of sulfur, nitrogen, and PAHs in the obtained products. Similar trends of properties of products from the hydrotreating of middle distillates were reported by Laredo et al.19 The products obtained by hydrotreating the winter blends had a higher content of sulfur, nitrogen, and PAHs than the corresponding products obtained by hydrotreating the summer blends, which was due to the higher content of these substances in the LCO-W compared with the LCO-S (Tables 2–4).

Only the products obtained by hydrotreating the summer feedstocks with the LCO content of up to 10 wt % and the products obtained by hydrotreating the winter raw materials with the LCO content of max 5 wt % met the requirements of EN 590 for the sulfur content. Feedstocks having a higher content of LCO would be necessary to desulfurize using severe conditions to obtain products with a sulfur content of max 10 mg·kg⁻¹, i.e., a higher temperature, a higher pressure, or a lower weight hourly space velocity would have to be used.

The viscosity of the hydrotreated products slightly decreased with increasing LCO content in the feedstock.
because the LCOs had lower viscosity than the SRGOs. All of the products and feedstocks had a viscosity value within the range of the EN 590 specification.

The density of the products also logically increased with increasing LCO content in the feedstock because the LCOs had a higher aromatic content and density than the SRGOs. Density is one of the properties that limits the LCO content in the feedstock. Only products with an LCO content in the raw materials up to 10 wt % fulfilled the requirement of EN 590 for density. Products prepared by hydrotreating a feedstock with more LCO would have to be mixed with kerosene. All of the products met the 8.0 wt % limit required by EN 590 for the PAH content (Tables 3 and 4).

All of the products had a cetane index greater than that specified in EN 590. The cetane index of the products decreased with increasing LCO content in the feedstock (Tables 3 and 4). An increase in the aromatic content and a related increase in the density and a decrease in the cetane index in hydrotreated products with an increasing LCO content in the feedstock were also observed by Laredo et al. and Ancheyta-Juárez et al. The cetane index of the products without a cetane improver is usually the same as their cetane number. The measured results of CI show that the cetane improver will need to be added to the products obtained by the hydrotreating of the feedstock containing 20 wt % LCO to gain products with the cetane number of at least 51 according to the EN 90 standard.

CFPP is a property mainly dependent on the content of long n-alkanes. The LCOs had a low saturated hydrocarbon content, they did not contain long n-alkanes and, hence, had a low CFPP. Therefore, the CFPP of the hydrotreated products slightly decreased with the increasing amount of LCO in the feedstocks (Tables 2–4). In the production of winter diesel, gas oil hydrotreatment products were blended with desulfurized kerosene and a depressant to meet the EN 590 requirement for the winter diesel fuel (F grade: −20 °C).

With the increasing LCO content in the feedstock, the content of PAHs in the obtained products increased (Tables 3 and 4). A greater decrease in the PAH content in the products compared to the raw materials was recorded for the raw materials with a high LCO content and, therefore, a high PAH content.

The group-type composition of the feedstocks and the products from their hydrotreatment is presented in Tables 5 and 6. It is clear from the presented data that the contents of triaromatics and diaromatics in the feedstock increased at the expense of saturated hydrocarbons with the increasing content of LCO in the feedstock. During the hydrotreatment of the feedstock, there was a significant decrease in the content of triaromatics and especially diaromatics, which were converted into monoaromatics and, to a lesser extent, into saturated hydrocarbons. The contents of diaromatics and triaromatics in the products increased depending on the increasing amount of LCO in the feedstock.

### 2.3. Hydrogen Consumption on Hydrotreating

The consumption of hydrogen on hydrotreating of the feedstocks was calculated on the basis of the mass balance and the composition of the feedstocks and the liquid products obtained by their hydrotreatment. It must be emphasized that the calculation of hydrogen consumption comes from...
Table 5. Group-Type Composition of the Summer Feedstocks and the Products from Their Hydrotreatment (wt %)

| feedstock/product | saturated hydrocarbons | monoaromatics | diaromatics | triaromatics |
|-------------------|------------------------|---------------|-------------|-------------|
| SF0               | 85.3                   | 8.5           | 5.6         | 0.6         |
| SF5               | 82.3                   | 8.7           | 8.2         | 0.9         |
| SF10              | 79.3                   | 8.8           | 10.8        | 1.2         |
| SF15              | 76.2                   | 9.0           | 13.3        | 1.4         |
| SF20              | 73.2                   | 9.1           | 15.9        | 1.7         |
| SH0               | 87.9                   | 10.2          | 1.8         | 0.1         |
| SH5               | 84.5                   | 12.8          | 2.6         | 0.1         |
| SH10              | 81.6                   | 14.8          | 3.4         | 0.2         |
| SH15              | 77.7                   | 17.1          | 4.7         | 0.4         |
| SH20              | 75.9                   | 19.0          | 4.9         | 0.3         |

Table 6. Group-Type Composition of the Winter Feedstocks and the Products from Their Hydrotreating (wt %)

| feedstock/product | saturated hydrocarbons | monoaromatics | diaromatics | triaromatics |
|-------------------|------------------------|---------------|-------------|-------------|
| WF0               | 85.1                   | 8.7           | 5.6         | 0.6         |
| WF5               | 82.0                   | 8.7           | 8.2         | 1.1         |
| WF10              | 78.9                   | 8.7           | 10.8        | 1.6         |
| WF15              | 75.8                   | 8.7           | 13.4        | 2.1         |
| WF20              | 72.7                   | 8.7           | 16.0        | 2.6         |
| WH0               | 86.7                   | 11.2          | 2.0         | 0.1         |
| WH5               | 84.1                   | 13.0          | 2.7         | 0.2         |
| WH10              | 80.5                   | 15.4          | 3.7         | 0.3         |
| WH15              | 77.7                   | 17.1          | 4.7         | 0.4         |
| WH20              | 74.0                   | 19.6          | 5.8         | 0.6         |

Table 7. Consumption of Hydrogen Related to 1 m³ of the Feedstock and the Excess of Hydrogen at the Inlet and Outlet of the Reactor

| feedstock | \(\text{V}_{\text{RF}}^{\text{HHDS}}\) [m³·h⁻¹] | \(\text{V}_{\text{RF}}^{\text{HHDN}}\) [m³·h⁻¹] | \(\text{V}_{\text{RF}}^{\text{HT}}\) [m³·h⁻¹] | \(\text{E}_{\text{RF}}^{\text{HHDS}}\) | \(\text{E}_{\text{RF}}^{\text{HHDN}}\) |
|-----------|---------------------------------------------|---------------------------------------------|---------------------------------------------|------------------------|------------------------|
| SF0       | 2.9                                         | 0.3                                         | 16.1                                        | 19.3                   | 12.5                   |
| SF5       | 3.2                                         | 0.4                                         | 20.0                                        | 23.6                   | 10.2                   |
| SF10      | 3.6                                         | 0.4                                         | 24.8                                        | 28.8                   | 8.3                    |
| SF15      | 3.9                                         | 0.5                                         | 25.8                                        | 30.2                   | 7.9                    |
| SF20      | 4.3                                         | 0.5                                         | 35.6                                        | 40.4                   | 5.9                    |
| WS0       | 2.9                                         | 0.2                                         | 13.8                                        | 17.0                   | 14.1                   |
| WS5       | 3.4                                         | 0.3                                         | 21.1                                        | 24.8                   | 9.7                    |
| WS10      | 3.8                                         | 0.4                                         | 25.2                                        | 29.4                   | 8.2                    |
| WS15      | 4.3                                         | 0.5                                         | 31.1                                        | 35.8                   | 6.7                    |
| WS20      | 4.8                                         | 0.5                                         | 34.8                                        | 40.0                   | 6.0                    |

The data in Table 7 show that hydrogen was consumed mainly for HDA and, to a lesser extent, for HDS of the feedstock, and the hydrogen consumption for HDN was very small. The hydrogen consumption increased with increasing LCO content in the feedstock, as the contents of sulfur, nitrogen, and especially diaromatics and triaromatics also increased with the increasing LCO content in the feedstock, and the amounts of sulfur, nitrogen, triaromatics, and diaromatics removed by hydrotreatment increased (Table 8). The amounts of sulfur, nitrogen, triaromatics, and diaromatics removed from the feedstock were calculated from the mass flow of these substances in the feedstock, from which the respective mass flow of these substances in the products was deducted, all in g·h⁻¹. The excess of hydrogen at the inlet and outlet of the reactor used for the hydrotreatment was large enough in all experiments performed. It is recommended to use a hydrogen-to-feedstock ratio that is three or four times higher than the expected hydrogen consumption to ensure an adequate hydrogen supply through the reactor. The excess of hydrogen decreased with increasing hydrogen consumption, which increased with increasing LCO content in the raw material.

2.4. Mixtures with Desulfurized Kerosene and/or Desulfurized Heavy Naphtha from FCC. Some products from the hydrotreatment were mixed with desulfurized kerosene and/or desulfurized heavy naphtha (HN) from the FCC. The properties of the feedstocks are listed in Table 9, and their distribution curve is shown in Figure 1. The kerosene and the heavy naphtha had a low temperature at which 95 vol % sample (T₉₅) was recovered, were deeply desulfurized, had a low PAH content, and a very good CFPP value. The heavy naphtha had a high density and a low cetane index (Table 9).

Mixture of SH15 and WH15 hydrotreated products with different kerosene contents were prepared. The properties of these mixtures are shown in Tables 10 and 11. The density was sufficiently improved to the EN S90 limit in the mixture containing 10 wt % kerosene. The density, viscosity, and cetane index of the blends decreased with increasing content of kerosene because kerosene had lower values of these properties than the hydrotreated products (Tables 3, 4, and 9).

The very low CFPP value of kerosene (Table 9) did not have a sufficient effect on the resulting CFPP values of the prepared mixtures (Tables 10 and 11).
Table 9. Properties of the Kerosene and Heavy Naphtha Used for the Blending

| feedstock                  | sulfur [mg·kg⁻¹] | nitrogen [mg·kg⁻¹] | monoaromatics [wt %] | PAHs [wt %] | density at 15 °C [kg·m⁻³] | viscosity at 40 °C [mm²·s⁻¹] | CI | CFPP [°C] | T95 [°C] |
|----------------------------|-----------------|-------------------|----------------------|-------------|---------------------------|-------------------------------|----|-----------|---------|
| kerosene                   | 1               | 16                | 14.8                 | 0.5         | 803                       | 1.3                          | 43 | <−40     | 221     |
| heavy naphtha              | 2               | 172               | 54.7                 | 5.1         | 865                       | 1.0                          | 17 | <−40     | 211     |

Table 10. Properties of the Mixture of the SH15 Product with Different Amounts of Kerosene

![Figure 1. Comparison of the distribution curves of kerosene and heavy naphtha (HN).](image)

Table 11. Properties of the Mixture of the WH15 Product with Different Amounts of Kerosene

![Figure 2. Dependency of the CFPP of the mixture of the WH15 product with 20 wt % kerosene on the depressant concentration.](image)

Table 12. Properties of the Mixtures of the SH10 Product with Different Amounts of Heavy Naphtha

| heavy naphtha in the mixture [wt %] | sulfur[a] [mg·kg⁻¹] | PAHs[a] [wt %] | density at 15 °C [kg·m⁻³] | viscosity at 40 °C [mm²·s⁻¹] | cetane index | CFPP [°C] |
|------------------------------------|---------------------|----------------|---------------------------|-------------------------------|--------------|-----------|
| 0                                  | 10                  | 3.6            | 843                       | 3.6                           | 55           | −6        |
| 5                                  | 10                  | 3.7            | 844                       | 3.3                           | 53           | −5        |
| 10                                 | 9                   | 3.8            | 845                       | 3.0                           | 51           | −5        |
| 15                                 | 9                   | 3.8            | 846                       | 2.8                           | 49           | −5        |

[a]Calculated values.

A higher reaction temperature to have a suitable sulfur content. The CFPP value of the mixtures with the heavy naphtha for the winter period would have to be reduced by the addition of a depressant. The other properties of the mixtures met the requirements of EN 590.

3. CONCLUSIONS

Straight-run gas oil (SRGO) and its mixtures with 5, 10, 15, and 20 wt % light cycle oil (LCO) from fluid catalytic cracking (FCC) were hydrotreated on a commercial NiMo/Al₂O₃ catalyst. The LCO had a high content of sulfur species that are difficult to desulfurize and a high density and low cetane index due to the high content of the bicyclic aromatic hydrocarbons. With the increasing content of LCO in the feedstock, the density and the contents of sulfur, nitrogen, and PAHs in the products increased, while their viscosity and cetane index decreased.

Only the products prepared from a raw material with an LCO content up to 10 wt % fulfilled the density requirement of EN 590. To use a larger amount of LCO, the products would have to be mixed with the desulfurized kerosene that has a lower density.

Only products obtained by hydrotreating the summer raw materials with an LCO content of up to 10 wt % and the...
products obtained by hydrotreating the winter raw materials with an LCO content of max 5 wt % met the requirements of EN 590 for the sulfur content. To obtain a product with the maximum sulfur content of 10 mg kg\(^{-1}\) from the feedstock with a higher LCO content, more severe conditions of hydrotreating would have to be used. To improve the density and CFPP of the hydrotreated products, mixtures of selected products with refined kerosene were prepared. The addition of kerosene decreased the density as expected, but the CFPP was only lowered by about 1–2 °C. It was necessary to add a depressant in an amount of 600 mg kg\(^{-1}\) to achieve the cold filter plugging point of −20 °C.

The use of desulfurized FCC heavy naphtha in the diesel fuel production was mainly limited by its high density. Up to 10 wt % heavy naphtha could be added to the product obtained by hydrotreating the raw material containing 10 wt % LCO. More than 15 wt % FCC heavy naphtha could be added to the mixture of 20 wt % kerosene and 80 wt % HW10 product. The blends prepared met with their density requirements of EN 590.

The hydrogen consumption increased with the increasing LCO content in the feedstock, as the contents of sulfur, nitrogen, and especially diaromatics and triaromatics also increased with the increasing LCO content in the feedstock, as the contents of sulfur, nitrogen, triaromatics, and diaromatics removed by hydrotreating increased.

### 4. EXPERIMENTAL SECTION

#### 4.1. Hydrotreating Process, Collection, and Stabilization of Products

Hydrotreatment of straight-run gas oil (SRGO) and its mixtures containing 5–20 wt % LCO was performed in a laboratory tubular reactor with the cocurrent flow of the raw material and hydrogen. The hydrotreatment was performed at a pressure of 4 MPa, a temperature of 350 °C, a constant weight hourly space velocity of ca. 1.0 h\(^{-1}\), and a hydrogen-to-feedstock ratio of 240 m\(^3\)·m\(^{-3}\).

The catalyst bed was filled with a 1:1 vol mixture of a commercial NiMo/Al\(_2\)O\(_3\) catalyst, whose particle size was adjusted to 0.25–0.42 mm, and an inert material (SiC, particle size from 0.25 to 0.30 mm). The total catalyst volume in the bed was 90 cm\(^3\) (93 g). The catalyst was activated following the process suggested by its manufacturer. A detailed description of the reactor, the feedstock, and the hydrogen feed has been given in a previous work.\(^{25}\)

The whole experiment lasted for about 150 h. Catalyst activation lasted for about 24 h, and it was followed by the stabilization of the catalyst activity, in which SRGO hydrotreating was performed for 24 h, and the sulfur content was measured every 4 h. When the sulfur content of the products did not change for at least 20 h, individual experiments were performed, each lasting for about 10 h. Setting and control of the reaction conditions took about 1 h, rinsing of the reactor and separator with reaction products took 6–8 h, and collection of the product took 2 h (200 mL of the liquid product was collected). After the last hydrotreatment conditions, the conditions from the first experiment were adjusted to verify the same catalyst activity at the beginning and the end of the experiment. Both samples showed the same level of desulfurization.

The collected liquid product was purged with hydrogen at a flow rate of 0.5 dm\(^3\)·min\(^{-1}\) for 2 h to remove the hydrogen sulfide. If the hydrogen sulfide remained in the product, it would be easily oxidized to elemental sulfur during further processing, which is undesirable in view of the evaluation of product desulfurization.

The stabilization of the liquid products from the hydrotreatment was carried out by distilling off the products boiling at 150 °C. The distillation was performed on a Fischer distillation apparatus equipped with an HSM 500 Spaltrohr column at a pressure of 5 kPa with a reflux ratio of 1:1. The gases exiting the column were frozen at −50 °C. The distillation was stopped when the temperature at the head of the column was 64 °C, which corresponds to 150 °C at atmospheric pressure.

#### 4.2. Analysis Methods

The density was determined according to EN ISO 12185 by a DMA 500 digital density meter (Anton Paar), and the kinematic viscosity was measured according to the ASTM D7042 using a Stabinger SVM 3000 viscometer. The cold filter plugging point (CFPP) was measured according to EN 116 using a CFPP apparatus (Normalab Analis, model 901).

For the improvement of the cold filter plugging point (CFPP), one type of a commercial flow improver (Infineum R288, Infineum UK Ltd) commonly used for the treatment of diesel fuels was used. The selected sample was treated with a flow improver at concentrations of 100, 300, and 600 mg kg\(^{-1}\).

The determination of the sulfur and nitrogen contents was performed using a Mitsubishi TOX-100 analyzer according to ASTM D5453 or ASTM D5762 and ASTM D4629, respectively. The content of the aromatic hydrocarbons was determined by high-performance liquid chromatography (HPLC) according to EN 12916.

The simulated distillation of the raw materials and the stabilized liquid products was performed according to ASTM D2887 on a TRACE GC (gas chromatograph). Conversion of the retention times on the boiling points was made using a calibration curve, which was obtained by the simulated distillation of a standard mixture of C\(_7\)–C\(_{30}\) n-alkanes with known boiling points. The detector signals were corrected for a blank so that the area under the distribution curve was equal to 100. The parameters of the simulated distillation have been listed in a previous work.\(^{27}\) The calculation of the cetane index was performed using the density and the data from the simulated distillation, which were converted to the distillation
curve according to ISO 3405. This conversion was provided according to ASTM D2887.

4.3. Calculation of Hydrogen Consumption. The consumption of hydrogen was calculated on the basis of the mass balance and the composition of the feedstocks used and the liquid products obtained by their hydrotreatment. The calculation made it possible to determine the approximate hydrogen consumption for the individual reactions taking place during hydrotreatment.

The mass flow of the individual components (sulfur, nitrogen, saturated compounds, monoaromatics, diaromatics, triaromatics) entering the reactor was calculated from the mass flow of the feedstocks and their composition according to eq 1.

\[ m_i^F = m_i^F \cdot w_i^F \]  
(1)

where \( m_i^F \) is the mass flow of the \( i \) component in the feedstock (g h\(^{-1}\)), \( m_i^F \) is the mass flow of the feedstock in g h\(^{-1}\), and \( w_i^F \) is the mass fraction of the \( i \) component in the feedstock.

The mass flow of the individual components contained in the stabilized liquid products was calculated according to eq 2.

\[ m_i^P = m_i^P \cdot w_i^P \]  
(2)

where \( m_i^P \) is the mass flow of component \( i \) contained in the stabilized liquid products in g h\(^{-1}\), \( m_i^P \) is the mass flow of the liquid product in g h\(^{-1}\), and \( w_i^P \) is the mass fraction of component \( i \) in the liquid product.

The hydrogen consumption for the hydrodesulfurization (HDS) of the feedstock (\( m_{\text{HDS}} \)) in g h\(^{-1}\) was calculated according to eq 3.

\[ m_{\text{HDS}} = (m_S^F - m_S^P) \cdot \frac{2M_{\text{H}}} {M_S} \]  
(3)

where \( m_S^F \) and \( m_S^P \) are the mass flows of sulfur in the feedstock and the appropriate product in g h\(^{-1}\), respectively; and \( M_{\text{H}} \) and \( M_S \) are the molar masses of molecular hydrogen and sulfur, respectively, both in g mol\(^{-1}\).

The calculation was performed with the estimation that two moles of hydrogen are consumed on average per mole of sulfur in the feedstock. This is true for the HDS of alkyl sulfides (Reaction RR1) and aryl sulfides (Reaction RR2) and direct and indirect HDS of alkyl thiophenes (Reactions RR3A, RR3D), alkyl benzothiophenes (Reactions RR4A, RR4D), and alkyl dibenzothiophenes (Reactions RR5A, RR5D), unless the hydrogenation of the double bonds in these compounds is considered. The hydrogen consumption for the hydrogenation of the double bonds or benzene rings in these compounds is considered. The hydrogen consumption for the hydrogenation of the unsaturated bonds resulting from the direct HDS (Reactions RR3B, RR4B) and eventual hydrogenation of the aromatic rings (Reaction RR5C), which occurs during the indirect HDS of the alkyl thiophenes, alkyl benzothiophenes, and alkyl dibenzothiophenes, is reflected in the group-type composition changes. Therefore, it is not included in the hydrogen consumption for the HDS but in the hydrogen consumption for the hydrodearomatization (HDA) that is shown below. For HDS of disulfides (Reaction RR6) and thiols (Reaction RR7), 3 and 1 moles of molecular hydrogen are needed, respectively.

The hydrogen consumption for the hydrodenitrogenation (HDN) of the feedstock (\( m_{\text{HDN}} \)) in g h\(^{-1}\) was calculated according to eq 4.

\[ m_{\text{HDN}} = (m_N^F - m_N^P) \cdot \frac{2M_{\text{H}}} {M_N} \]  
(4)

where \( m_N^F \) and \( m_N^P \) are the mass flows of nitrogen in the feedstock and the appropriate product in g h\(^{-1}\), respectively, and \( M_N \) is the molar mass of the nitrogen atom in g mol\(^{-1}\).

The calculation was performed with the estimation that two moles of hydrogen are consumed on average per mole of nitrogen in the feedstock. Two moles of hydrogen are consumed for the HDN of the alkyl pyrroles (Reaction 8B), alkyl indoles (Reaction RR9B), and alkyl carbazoles (Reaction RR10B), unless the hydrogenation of the double bonds in these compounds is considered (Reactions RR8A, RR9A, RR10A). One mole of hydrogen is consumed for the HDN of the alkyl pyrroles (Reaction 8B), nitrobenzene (Reaction RR11), and aryl amines (Reaction RR12). Two moles of hydrogen are needed for the HDN of the alkyl amines (Reaction RR13B), alkyl quinolines (Reaction RR14B), alkyl benzoquinolines (Reaction RR15B), and alkyl acridines (Reaction RR16B), unless the hydrogenation of the double bonds is considered. The hydrogen consumption for the hydrogenation of the double bonds (Reactions RR8A, RR9A, RR10A, 13A, RR14A, RR15A, RR16A) and eventual hydrogenation of the aromatic rings of the nitrogen compounds, which occurs during the HDN of some nitrogen compounds, have an impact on the changes in the group-type composition; therefore, it was included in the hydrogen consumption for the HDA.
The amount of triaromatics removed by the HDA ($m_{TA}^{DA}$) is approximately equal to the amount of diaromatics formed from them in g·h$^{-1}$ ($m_{DA}^{DA}$) (eq 7), which have to be considered when calculating the hydrogen consumption to convert the diaromatics into monoaromatics, which was calculated according to eq 8.

$$m_{TA}^{DA} \cong m_{TA}^{Re} = (m_{TA}^{F} - m_{TA}^{P})$$

$$m_{DA}^{DA} = (m_{DA}^{F} + m_{DA}^{TA} - m_{DA}^{P}) \cdot \frac{2M_{H2}}{M_{DA}^{ST}}$$

where $M_{DA}^{ST}$ is the molar mass of the hexyl naphthalene ($m_{DA}^{ST} = 212$ g·mol$^{-1}$), which was chosen as the diaromatic standard for the calculation, and $m_{DA}^{F}$ and $m_{DA}^{P}$ are the mass flows of the diaromatics in the feedstock and the related stabilized products in g·h$^{-1}$, respectively.

The amount of monoaromatics formed by the hydrogenation of the diaromatics ($m_{MA}^{DA}$) is approximately equal to the amount of diaromatics removed during the hydrotreatment ($m_{DA}^{Re}$) in g·h$^{-1}$ (eq 9), which have to be taken into account when calculating the hydrogen consumption needed to convert part of the monoaromatics into saturated hydrocarbons. It was calculated according to eq 10.

$$m_{MA}^{DA} = (m_{DA}^{F} + m_{DA}^{TA} - m_{DA}^{P}) \cdot \frac{3M_{H2}}{M_{MA}^{ST}}$$

where $M_{MA}^{ST}$ is the molar mass of the C12 alkyl benzene ($m_{MA}^{ST} = 246$ g·mol$^{-1}$), which was chosen as the monoaromatic standard for the calculation, and $m_{MA}^{F}$ and $m_{MA}^{P}$ are the mass flows of the monoaromatics in the feedstock and the stabilized products in g·h$^{-1}$, respectively.

The total hydrogen consumption for the hydrotreatment of the feedstock ($m_{H_{2}}^{In}$) in g·h$^{-1}$ was calculated according to eq 11.

$$m_{H_{2}}^{In} = m_{H_{2}}^{Re} + m_{H_{2}}^{RDS} + m_{H_{2}}^{HDA}$$

The excess of hydrogen relative to its consumption varies along the reactor depending on hydrogen consumption. The excess of hydrogen relative to its consumption at the reactor inlet ($E_{H_{2}}^{In}$) was calculated according to eq 12.

$$E_{H_{2}}^{In} = m_{H_{2}}^{F} / m_{H_{2}}^{In}$$

where $m_{H_{2}}^{F}$ is the hydrogen mass flow used for the hydrotreatment in g·h$^{-1}$.

The total volume of hydrogen consumed on hydrotreatment related to 1 m³ of the feedstock ($V_{H_{2}}^{RF}$) in m³·m⁻³ was calculated according to eq 14.

$$V_{H_{2}}^{RF} = m_{H_{2}}^{F} \cdot M_{H_{2}}^{M} / (M_{H_{2}}^{In} \cdot V_{H_{2}}^{RF})$$

where $V_{H_{2}}^{M}$ is the hydrogen molar volume at 20 °C in dm³·mol⁻¹, $M_{H_{2}}^{M}$ is the hydrogen (H₂) molar mass in g·mol⁻¹, and $V_{H_{2}}^{RF}$ is the feedstock volume flow in dm³·h⁻¹.

Similarly, the volume of hydrogen consumed on HDS, HDN, and HDA related to 1 m³ of the feedstock in m³·m⁻³.
(\(V_{HF}^{RF}, V_{HF}^{HC},\) and \(V_{HF}^{DA}\), respectively) was calculated according to eqs 15–17.

\[ V_{HF}^{RF} = m_{HF}^{RF} \frac{V_{M}^{RF}}{(M_{H_2}^{RF})} \]  
\[ V_{HF}^{HC} = m_{HF}^{HC} \frac{V_{M}^{HC}}{(M_{H_2}^{HC})} \]  
\[ V_{HF}^{DA} = m_{HF}^{DA} \frac{V_{M}^{DA}}{(M_{H_2}^{DA})} \]  

\[ (\text{15}) \]
\[ (\text{16}) \]
\[ (\text{17}) \]

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\section*{Notes}
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\section*{ABBREVIATIONS USED}

- BTX: benzene, toluene, and xylenes
- CFPP: cold filter plugging point
- CI: cetane index
- LCO: light cycle oil
- FCC: fluid catalytic cracking
- HDA: hydrodearomatization
- HDN: hydronitrogenation
- HDS: hydrodesulfurization
- HN: heavy naphtha
- PAHs: polycyclic aromatic hydrocarbons
- R250: amount of distillate recovered at 250 °C
- SRGO: straight-run gas oil
- T95: temperature at which 95 vol % sample was recovered

\[ \text{Ex}_{\text{Out}} \] excess of hydrogen relative to its consumption at the reactor outlet
\[ \text{Ex}_{\text{In}} \] excess of hydrogen relative to its consumption at the reactor inlet

\[ m_{\text{F}}^{*} \] mass flow of the feedstock in g·h\(^{-1}\)
\[ m_{\text{P}}^{*} \] mass flow of the liquid product in g·h\(^{-1}\)

\[ m_{H}^{*} \] hydrogen mass flow used for the hydrotreatment in g·h\(^{-1}\)

\[ m_{\text{F}}^{*} \] mass flow of component \( i \) in the feedstock in g·h\(^{-1}\)

\[ m_{\text{P}}^{*} \] mass flow of component \( i \) contained in the stabilized liquid products in g·h\(^{-1}\)

\[ m_{\text{S}}^{*} \] mass flow of nitrogen in the feedstock in g·h\(^{-1}\)

\[ m_{\text{L}}^{*} \] mass flow of sulfur in the feedstock in g·h\(^{-1}\)

\[ m_{\text{D}}^{*} \] mass flow of sulfur in the liquid product in g·h\(^{-1}\)

\[ m_{\text{F}}^{*} \] mass flow of diaromatics in the feedstock in g·h\(^{-1}\)

\[ m_{\text{P}}^{*} \] mass flow of diaromatics in the stabilized products in g·h\(^{-1}\)

\[ m_{\text{r}}^{*} \] amount of diaromatics removed during the hydrotreating in g·h\(^{-1}\)

\[ m_{\text{r}}^{*} \] amount of diaromatics formed by triaromatics hydrogenation in g·h\(^{-1}\)

\[ m_{H}^{*} \] total hydrogen consumption for the hydrotreatment of the feedstock (\( \cdot \)) in g·h\(^{-1}\)

\[ m_{\text{r}}^{*} \] hydrogen consumption to convert the diaromatics to monoaromatics in g·h\(^{-1}\)

\[ m_{\text{r}}^{*} \] hydrogen consumption to convert the monoaromatics to saturated hydrocarbons in g·h\(^{-1}\)

\[ m_{H}^{*} \] hydrogen consumption to convert the triaromatics to diaromatics in g·h\(^{-1}\)

\[ m_{H}^{*} \] hydrogen consumption for the HDN of the feedstock in g·h\(^{-1}\)

\[ m_{H}^{*} \] hydrogen consumption for the HDS of the feedstock in g·h\(^{-1}\)

\[ m_{\text{r}}^{*} \] mass flow of monoaromatics in the feedstock
\[ m_{\text{r}}^{*} \] mass flow of monoaromatics in the stabilized products in g·h\(^{-1}\)

\[ m_{\text{r}}^{*} \] amount of monoaromatics formed by the hydrogenation of the diaromatics
\[ m_{\text{r}}^{*} \] mass flow of triaromatics in the feedstock in g·h\(^{-1}\)

\[ m_{\text{r}}^{*} \] mass flow of triaromatics in the stabilized products in g·h\(^{-1}\)

\[ m_{\text{r}}^{*} \] amount of triaromatics removed by the HDA in g·h\(^{-1}\)

\[ M_{\text{H}}^{*} \] molar mass of molecular hydrogen in g·mol\(^{-1}\)

\[ M_{\text{N}}^{*} \] molar mass of the nitrogen atom in g·mol\(^{-1}\)

\[ M_{\text{S}}^{*} \] molar mass of sulfur in g·mol\(^{-1}\)

\[ M_{\text{T}}^{*} \] molar mass of the diaromatic standard (hexyl naphthalene, \( m_{\text{T}}^{*} = 212 \) g·mol\(^{-1}\))

\[ M_{\text{MA}}^{*} \] molar mass of the monoaromatic standard (\( C_{12} \) alkyl benzene, \( m_{\text{MA}}^{*} = 246 \) g·mol\(^{-1}\))

\[ M_{\text{T}}^{*} \] molar mass of the triaromatic standard (phenanthrene, \( m_{\text{T}}^{*} = 178 \) g·mol\(^{-1}\))

\[ V_{\text{F}}^{*} \] feedstock volume flow in dm\(^3\)·h\(^{-1}\)

\[ V_{\text{HF}}^{HC} \] volume of hydrogen consumed on HDA related to 1 m\(^3\) of the feedstock in m\(^3\)·m\(^{-3}\)

\[ V_{\text{HF}}^{RF} \] volume of hydrogen consumed on HDN related to 1 m\(^3\) of the feedstock in m\(^3\)·m\(^{-3}\)

\[ V_{\text{HF}}^{RF} \] volume of hydrogen consumed on HDN related to 1 m\(^3\) of the feedstock in m\(^3\)·m\(^{-3}\)

\[ V_{\text{HT}}^{RF} \] total volume of hydrogen consumed on hydrotreating related to 1 m\(^3\) of the feedstock in m\(^3\)·m\(^{-3}\)

\[ w_{\text{F}}^{*} \] mass fraction of component \( i \) in the liquid product

\[ w_{\text{P}}^{*} \] mass fraction of the component \( i \) in the feedstock

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