

HYDROCRACKING OF FISCHER-TROPSCH WAX

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The low-temperature Fischer-Tropsch synthesis (LTFT) processing of renewable feedstocks combined with the hydrocracking of its solid product is an effective way to produce synthetic renewable engine fuels. The hydrocracking of an FT wax derived from natural gas using the LTFT synthesis was studied in this paper. The hydrocracking was carried out in a tubular fixed-bed reactor with a co-current flow of the feedstock and hydrogen. Reaction temperatures in the range of 305–370 °C, a pressure of 8 MPa, an H₂/feed ratio of 500 m³/m³ and weight hour space velocities (WHSV) of 1; 2 and 4 h⁻¹ were tested. The naphtha fraction (boiling up to 200 °C) was the main product of the hydrocracking under all the tested reaction conditions. It could be used as a component into petroleum-derived gasoline in a neat form or the after processing by common refinery processes (isomerization and/or reforming). The production of low-sulfur and low-aromatic paraffinic solvent or the utilization as a feedstock for steam cracking could be some other options of the naphtha fraction utilization. The maximum yield of the gaseous products (depending on the reaction temperatures and WHSV) was 20 wt.%. They were primarily composed of n-alkanes and isoalkanes and could be, therefore, used as an optimal feedstock for steam cracking as well. The C₃-C₄ fraction of the gaseous products could be also utilized as an LPG fuel. Very low yields (up to 10.4 wt.%) of the middle distillates were obtained under all the tested reaction conditions. Due to their saturated nature, their densities were very low and, additionally, poor low-temperature properties can be expected.

Keywords: Fischer-Tropsch synthesis, Hydrocracking, FT Wax, Synthetic fuels

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1. Introduction

In recent years, the production of liquid fuels from various sources (coal, natural gas, biomass) via the Fischer-Tropsch synthesis (FTS) process has gained renewed significance. The FTS is recognized as an industrially proven and economically competitive route to produce high-quality engine fuels. At the present time, special attention is paid to fuels made from waste biomass, because these advanced biofuels may contribute to greenhouse gas emissions reduction. FTS is a well-known and established catalytic chemical process, which was named after two German inventors, Franz Fischer and Hans Tropsch in the 1920s. During World War II, FTS was an essential source of liquid hydrocarbon fuels and necessary chemicals for the German war effort. Since then, many modifications and adjustments of the technology have been made, including catalyst and reactor design development [1–3].

Generally, the technology producing liquid hydrocarbon fuels from non-petroleum raw materials can be referred to as an XTL process (x-to-liquids) [4]. The main part of the XTL process is the Fischer-Tropsch synthesis process, which converts synthesis gas (a mixture of carbon monoxide and hydrogen) into hydrocarbons, which are then processed into fuels and petrochemical feedstocks [5]. The base material for the production of the synthesis gas can be any organic carbonaceous material. Depending on its type, the technology is referred to as coal-to-liquids (CTL), gas-to-liquids (GTL) or biomass-

to-liquids (BTL). The FT wax utilized in this work was produced via the GTL process which is the most widespread process of the FTS technologies and consists of three stages. In the first stage, natural gas is partially oxidized in order to create a mixture of hydrogen and carbon monoxide, which is known as synthesis gas or syngas. In the second stage (after the syngas composition adjustment), the synthesis gas is converted into saturated hydrocarbons using a catalyst. The heaviest fraction of the obtained product is called “syncrude” and is commonly solid at room temperature. The final stage of the XTL process is based on the cracking and isomerization of the syncrude, which converts long hydrocarbons into shorter ones. This process yields high-quality liquid products such as diesel, kerosene and/or lubricant oil. These products are colorless, odorless low-aromatic sulfur-free liquids of high quality and contain a very low level of impurities [6–9].

In summary, a syncrude can be considered as a hydrocarbon-based mixture with a wide range of molecular weights, whose distribution can be described by the Anderson-Schulz-Flory model [10]. In comparison with petroleum-based materials, syncrude does not contain any nitrogen or sulfur compounds, but it may contain a small amount of oxygen compounds, such as alcohols and carboxylic acids. The composition of the products varies depending on many factors, such as the reaction temperature, reaction pressure, type of reactor and type of catalyst. On the other hand, FTS is almost independent to the

origin of the raw material. Similar results regarding yields and composition can, thus, be expected from the CTL, GTL and BTL processes.

In principle, FTS can be divided into two categories: low-temperature (LTFT) and high-temperature (HTFT). LTFT is commonly operated in a temperature range of 200–250 °C, a pressure range of 2–3 MPa using a catalyst based on iron or cobalt. HTFT is usually operated at temperatures of 300–350 °C, pressures of 1.5–3 MPa using a catalyst based on iron.

Reaction conditions of LTFT are appropriate for the production of high molecular weight paraffins called waxes (the substances with a boiling point higher than 370 °C) [11,12]. These waxes can be afterwards processed via hydrocracking over a bifunctional catalyst in order to produce high-quality engine fuels [13].

The hydrocracking process is usually aimed at the high selectivity of the heavy paraffin conversion into hydrocarbons boiling in the range of 200–400 °C and, simultaneously, it should minimize its consecutive cracking into gaseous products and naphtha. Beyond this, the isomerization of the linear alkanes should be supported to improve the low-temperature properties of the middle distillates. Hydrocracking bifunctional catalysts seem to be the most suitable for this purpose due to the presence of acidic sites that provide cracking and isomerization functions, and of metal sites with a hydrogenation-dehydrogenation function. The optimal reaction pressures and temperatures of the FT wax hydrocracking are in the range of 3–7 MPa and 320–370 °C, respectively [14-17]. The possibility of using milder reaction conditions and a higher WHSV compared to the hydrocracking of petroleum-based feedstocks (e.g., vacuum gas oil) can be explained by the nature of the FT waxes, *i.e.* the high chemical reactivity of the long paraffinic molecules and the absence of the hardly treatable compounds. The milder process conditions allow one to achieve high conversion and good catalyst stability. Hydrocracking of the LTFT waxes is a valuable option to produce high-quality liquid engine fuels, especially middle distillates. The proper choice of the operating conditions and catalyst type are crucial for the optimal result of the hydrocracking process.

In this paper, the attention was focused on the hydrocracking of the FT wax and the detailed evaluation of the obtained gaseous and liquid products. The composition and basic physicochemical properties of the obtained distillates were evaluated and compared with the current standard specifications for engine fuels (EN 228 for gasoline and EN 590 for diesel fuel) [18,19].

2. Experimental setup

2.1. Characteristics of the feedstock

A paraffinic FT wax Sarawax SX70 (Shell, Malaysia) was used as the feedstock in this study. It was produced via the GTL technology and was composed of a 98.8 wt.% of a fraction boiling above 400 °C and of a 1.2 wt.% of a fraction boiling in the range of 200–400 °C. Its basic physicochemical parameters are listed in Tab. 1.

Tab. 1 The physicochemical parameters of Sarawax SX70

Parameter	Value	Method
Density at 15 °C (kg·m ⁻³)	842	ASTM D1217
Density at 70 °C (kg·m ⁻³)	780	DIN 51757
Congealing point (°C)	72	ASTM D938
Kin. viscosity at 100 °C (mm ² ·s ⁻¹)	6.5	ASTM D445
Simulated distillation		ISO 3924
	T10	438
	T50	488
	T90	535
Sulfur content (mg·kg ⁻¹)	< 1	ISO 20846
Carbonization residue (wt.%)	0.005	ISO 10370
Appearance	white granules	Visual

2.2. Catalytic hydrocracking

The FT wax hydrocracking experiments were carried out via a continuous-flow catalytic unit with a fixed-bed reactor and a co-current flow of the feedstock and hydrogen. The reactor consisted of a stainless-steel tube with an internal diameter of 30 mm and a length of 620 mm. The reactor was loaded with 43.7 g of a catalyst with a particle size range of 0.25–0.42 mm. The utilized catalyst was a typical hydrocracking catalyst based on Ni, W and Mo/zeolite Y + Al₂O₃. The catalyst was diluted with silicon carbide (SiC) at a ratio of 1:1 (V:V). The catalyst was, at first, activated (sulfided) using hydrotreated gas oil (HGO) containing 3 wt.% of dimethyl disulfide and afterwards stabilized using straight run gas oil (SRGO). Dibutyl sulfide (DBS) was added in the concentration of 1 wt.% to the FT wax to maintain the activity of the sulfidic catalyst during the hydrocracking process. The reaction conditions of the FT wax hydrocracking are summarized in Tab. 2. Reaction temperatures in the range of 305–370 °C, a pressure of 8 MPa, an H₂/feed ratio of 500 m³/m³ and a weight hour space velocity (WHSV) of 1; 2 and 4 h⁻¹ were tested.

Tab. 2 The reaction conditions of the hydrocracking

WHSV (h ⁻¹)	Reaction Temperature (°C)	Pressure (MPa)	H ₂ /feed ratio (m ³ ·m ⁻³)	Feedstock flowrate (g·h ⁻¹)
1	305; 310; 315; 320; 325; 330; 340	8	500	45
2	330; 340; 350; 360	8	500	90
4	330; 340; 350; 360; 370	8	500	180

2.3. Analysis of the products

Liquid and gaseous hydrocracking products were collected for each combination of reaction conditions (see Tab. 2). The composition of the gaseous products was determined using gas chromatography with a flame ionization detector equipped with a quartz capillary column HP-PLOT (50 m length \times 0.3 mm (ID) \times 0.8 μ m film thickness).

The primary liquid products were, at first, analyzed using simulated distillation (SIMDIST) and consequently fractionated by column distillation into a naphtha fraction (boiling up to 200 °C), a middle distillate (200–400 °C) and a residue (above 400 °C). The column distillation was performed via a FISCHER distillation apparatus with a SPALTROHRTH HMS 500 spiral column with 90 theoretical trays at the following parameters: a reflux of 1:1, a maximum temperature in the distillation flask of 300 °C and a maximum temperature on the head of the distillation column of 200 °C.

The obtained naphtha fractions were characterized by the density, dry vapor pressure equivalent (DVPE), distillation test and GC-FID PONA analysis (see Tab. 3).

The kinematic viscosity, density, HPLC and cetane index were used for the characterization of the middle distillates (see Tab. 4). The simulated distillation and ASTM D2887 method were used to obtain the values required for the cetane index calculation. The obtained results were compared with the EN 228 and EN 590 standard specifications for gasoline and diesel fuel, respectively [18,19].

3. Results and discussion

3.1. Mass balance of the hydrocracking

The conversion, the yields of the gaseous and liquid hydrocracking products as well as the fractional composition of the liquid products are summarized in Tab. 5. The reported yields are related to the feedstock used for the hydrocracking. All the samples were labeled this way: reaction temperature in °C/WHSV in h^{-1} .

The yields of the liquid product ranged between 73 and 92 wt.% and increased with an increasing WHSV. On the other hand, an increase in the reaction temperature resulted in a reduction of the liquid product yields. The conversion, i.e., the transformation of the fraction boiling above 400 °C into liquid products boiling below 400 °C, was calculated from the results of the liquid products fractionation (column distillation). The conversion increased with an increasing reaction temperature and decreased with an increasing WHSV. The full conversion was reached at a temperature of 315 °C for a WHSV of 1 h^{-1} , at 340 °C for a WHSV of 2 h^{-1} and at 350 °C for a WHSV of 4 h^{-1} . The reaction conditions needed for the full conversion are significantly milder when compared to the hydrocracking of petroleum vacuum distillates where a minimal temperature of around 380 °C, a pressure above 16 MPa and a WHSV below 1 h^{-1} are required [20].

Tab. 3 The basic parameters used for the characterization of the naphtha fractions and the EN 228 specification for the petrol/gasoline

Parameter	Limits		Testing methods
	Min.	Max.	
Density at 15 °C ($\text{kg}\cdot\text{m}^{-3}$)	720.0	775.0	EN ISO 12185
Vapor pressure (kPa)			EN 13016-1
class A (summer)	45.0	60.0	
class D (winter)	60.0	90.0	
Distillation test (vol. %)			
E70 class A	20.0	48.0	
class D	22.0	50.0	EN ISO 3405
E100	46.0	71.0	
E150	75.0	-	
FBP (°C)	-	210	
Aromatics (vol.%)	-	35.0	EN ISO 22854
Benzene (vol.%)	-	1.00	EN ISO 22854
Olefins (vol.%)	-	18.0	EN ISO 22854

Tab. 4 The basic parameters used for the characterization of the middle distillates and the EN 590 specification for the automotive diesel fuel

Parameter	Limits		Testing methods
	Min.	Max.	
Kinematic viscosity at 40 °C ($\text{mm}^2\cdot\text{s}^{-1}$)	2.00	4.50	EN ISO 3104
Density at 15 °C ($\text{kg}\cdot\text{m}^{-3}$)	820.0	845.0	EN ISO 12185
Polycyclic aromatic hydrocarbons (wt. %)	-	8.0	EN 12916
Cetane index	46.0	-	EN ISO 4264

It is evident (see Tab. 5) that the catalyst's selectivity for the middle distillates (200–400 °C) was very low at all tested reaction temperatures. In comparison with the other research projects, where the yields of middle distillates varied around 60 wt.% [15-17], the yields in this project were much lower. It can be mainly attributed with the used catalyst because the other reaction conditions utilized in this project were similar to the others. The yield of the middle distillates varied between 0 and 10.4 wt.% and the wax was, thus, easily hydrocracked mainly into the naphtha fraction and the gaseous products.

3.2. Gaseous products

The gaseous hydrocracking products were predominantly composed of n-alkanes and isoalkanes and their concentrations varied in the range of 87–289 $\text{g}\cdot\text{m}^{-3}$ and 153–317 $\text{g}\cdot\text{m}^{-3}$, respectively. The content of the other hydrocarbons groups was negligible.

Tab. 5 The mass balance of the hydrocracking and the yields of the obtained products

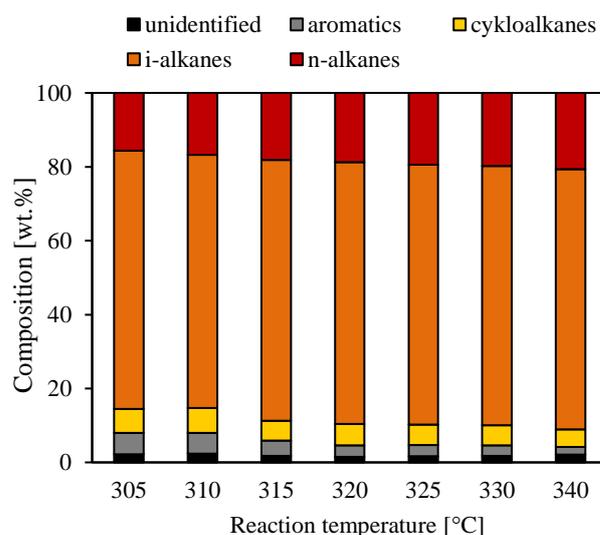
Sample (temp./WHSV)	Yields of hydrocracking products (wt.%)			Yields of fractions (wt.%) – related to the feedstock				Conversion (%)
	Products		Losses	< 200 °C	200–400 °C	> 400 °C	Losses	
	Liquid	Gaseous						
305/1	80.9	15.4	3.7	37.4	9.9	32.2	1.3	67.4
310/1	80.1	15.9	4.0	40.0	10.4	27.9	1.3	71.7
315/1	79.2	18.1	2.7	66.9	9.0	-	2.6	100
320/1	78.4	19.8	1.8	67.3	7.8	-	2.7	100
325/1	76.9	20.0	3.1	70.4	3.7	-	2.0	100
330/1	75.3	20.7	4.0	67.1	4.6	-	3.4	100
340/1	73.3	21.4	5.3	66.5	2.2	-	3.9	100
330/2	87.0	7.7	5.3	67.1	9.4	9.4	1.0	90.5
340/2	84.5	9.7	5.8	76.0	5.2	-	4.0	100
350/2	81.2	13.3	5.5	76.2	1.4	-	3.3	100
360/2	79.7	16.6	3.7	78.9	-	-	-	100
330/4	91.7	2.8	5.5	30.6	8.9	50.4	1.0	51.1
340/4	90.8	5.8	3.4	50.0	8.5	30.9	1.3	65.5
350/4	89.0	6.0	5.1	80.4	7.5	-	1.1	100
360/4	88.9	6.8	4.3	83.2	3.3	-	1.7	100
370/4	87.1	8.7	4.2	83.8	0.8	-	2.1	100

The concentration of the hydrocarbons, especially propane and butanes increased with an increasing reaction temperature. The concentration of the higher hydrocarbons decreased with the reaction temperature. Increasing the WHSV led to the reduction of the yield in the gaseous products. Gaseous products can be used in the same way as similar petroleum-derived products. They can be used for LPG production or as a feedstock for steam cracking. Although the FT wax used in this work was not produced from a biomass, the same results can be expected with such a feedstock. Such gaseous products can, thus, contribute not only to the production of renewable LPG fuel with low GHG emissions, but also to the production of renewable (“green”) monomers for the petrochemical industry.

3.3. Naphtha fraction

The results of the group composition of the naphtha fractions obtained at WHSV 1 h⁻¹ measured by GC-FID analysis are shown in Fig. 1. It is evident that an increasing reaction temperature led to a slight increase in the n-alkanes content and a decrease in the cycloalkanes and aromatics content. The content of the isoalkanes, as the main component of the naphtha fractions, was not significantly affected by the reaction temperature. Similar trends were observed for the naphtha obtained at WHSV 2 and 4 h⁻¹ as well. An increasing WHSV caused an increase in the content of the cycloalkanes and aromatic hydrocarbons, especially at lower reaction temperatures (see fig. 2). It can be explained by increasing hydrogenation activity of the catalyst at higher temperatures and lower WHSV. Nevertheless, all the samples complied with the

requirements of the EN 228 specification regarding the aromatics content (not exceeding 7.4 vol.%), the benzene content (not exceeding 0.68 vol.%) and the olefins content (not detected) with a large margin. Due to the rather high content of n-alkanes and the low content of aromatics in all the naphtha fractions, values of the research octane number (RON) significantly lower than 95 units, i.e., the lower limit of the EN 228 specification, can be expected. The estimation of the RON, based on the calculations from the GC-FID analysis providing the detailed hydrocarbon analysis was in the range of 65-80 units.

**Fig. 1** The results of the GC-FID analysis of the naphtha fractions for WHSV 1 h⁻¹

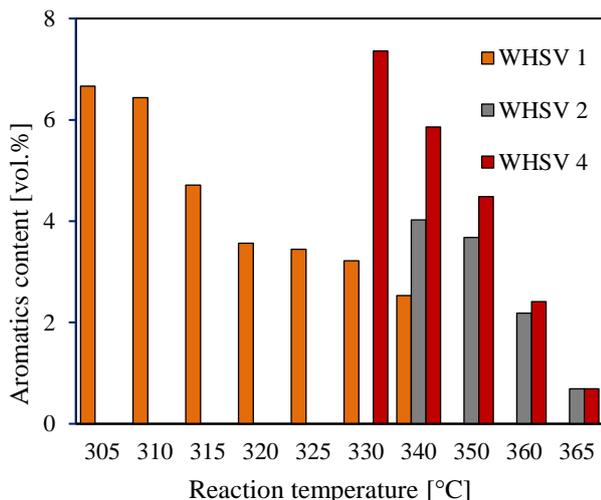


Fig. 2 The aromatics content in the naphtha fractions

The densities of all the gasoline samples were lower than the lower limit required by the EN 228 specification as is evident in Fig. 3. Moreover, an increasing reaction temperature and decreasing WHSV were followed by a density decrease, which is associated with a decrease in the cycloalkanes and aromatics content.

The DVPE of the analyzed naphtha fractions increased with an increasing reaction temperature (see Fig. 4). The use of the higher WHSV was reflected by a reduction in the DVPE. Most of the analyzed samples met the requirements that EN 228 put on gasoline class D (winter type). The DVPE of samples 305/1; 310/1; 330/4 and 340/4 was lower than the minimum value required for gasoline class A (summer type).

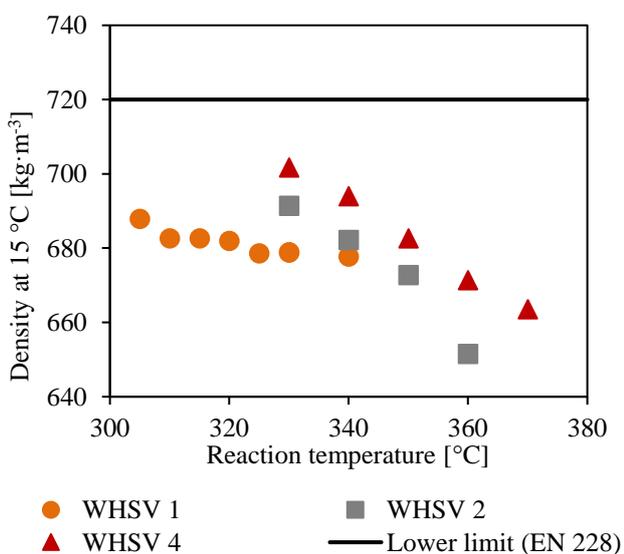


Fig. 3 The density of the naphtha fractions

The results of the distillation test are presented in Figs. 5, 6 and 7. The increasing reaction temperature was reflected by an increase in the E70, E100 and E150 parameters and a decrease in the final boiling point (FBP) during the distillation test.

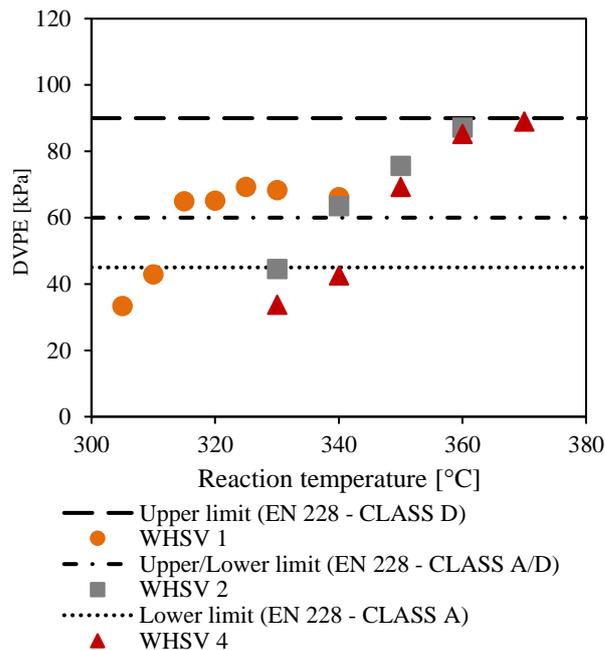


Fig. 4 The vapor pressure of the naphtha fractions

Using the higher WHSV led to a decrease in the E70, E100 and E150 parameters and an increase in the FBP. Most of the samples met the requirements of the EN 228 specification regarding all the parameters of the distillation test. Samples 360/2 and 370/4 were too volatile, so the requirements put on the E70 and E100 parameters were not satisfied. On the other hand, the E70 and E100 parameters of the samples 305/1; 310/1; 330/2 and 340/4 were lower than the lower limit required by the specification.

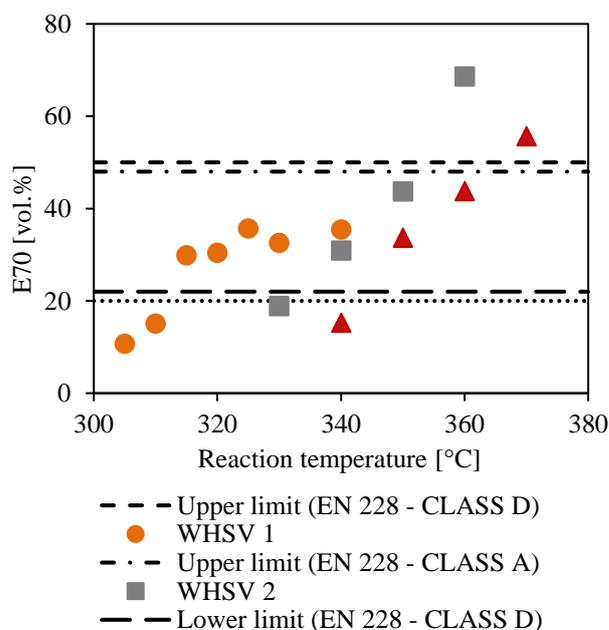


Fig. 5 E70 of the naphtha fractions

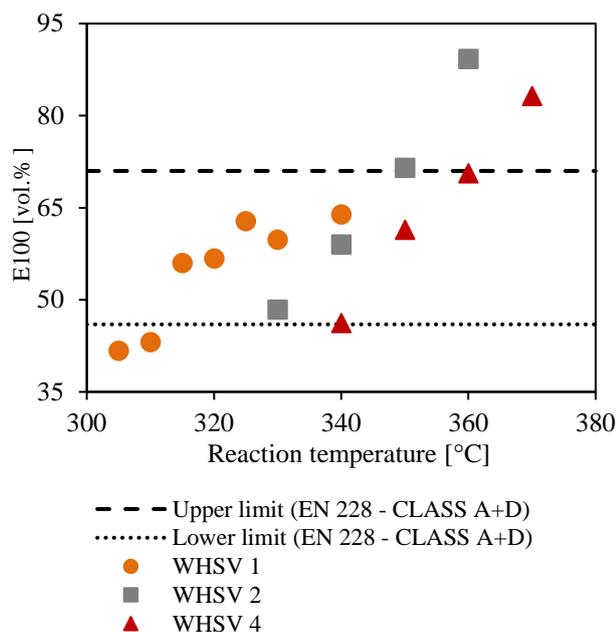


Fig. 6 E100 of the naphtha fractions

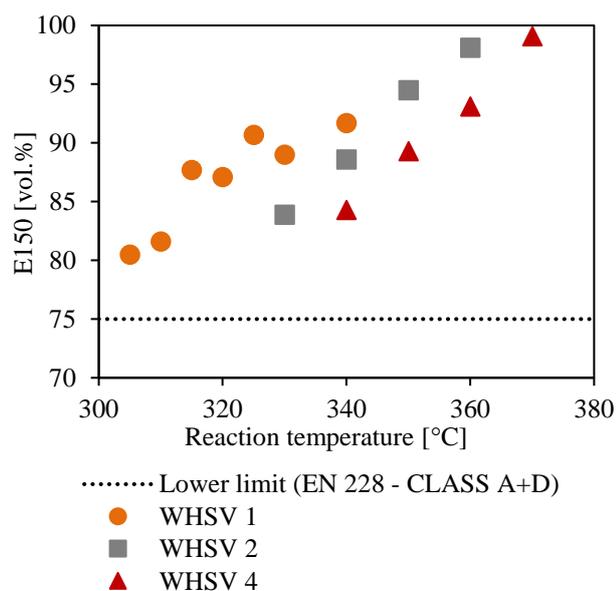


Fig. 7 E150 of the naphtha fractions

Generally, all the tested naphtha fractions could be used as a blending component for the production of automotive gasoline in limited quantities. Such a utilization can be likened to the utilization of petroleum-derived naphtha from hydrocracking or straight-run naphtha from the distillation of petroleum. The whole production of such naphtha could be used for gasoline blending after processing by common refinery technologies (isomerization and reforming). The production of special sulfur-free low-aromatic solvents or the utilization as a high-quality feedstock for steam cracking is also possible. The naphtha fractions produced by BTL process can, thus, become a renewable (advanced biofuel) component of automotive gasoline fully compatible with petroleum-derived

gasoline and refinery technologies. Besides that, they can be used as a valuable renewable feedstock for the petrochemical industry.

3.4. Middle distillates

The results of the middle distillates characterization are presented in Tab. 6. Since the yields of the middle distillates were generally rather low, only several parameters were determined. All the analyzed samples were composed of at least 97 wt.% of saturated hydrocarbons, while the remaining part was predominantly formed by monocyclic aromatic hydrocarbons. The content of the polycyclic aromatic hydrocarbons (PAH), i.e., compounds with two or more aromatic rings, was negligible in all the middle distillates. All the samples, therefore, met the requirements of the EN 590 specification. The calculated cetane index of all the samples, for which all the necessary data were available, was higher than 70. This value is much higher than the lower limit required by the EN 590 specification. The densities of all the analyzed samples were much lower than the lower limit required by the specification. The highest measured density was $778.3 \text{ kg}\cdot\text{m}^{-3}$ for the 305/1 sample which is significantly below the lower limit of the EN 590 specification, i.e., $820 \text{ kg}\cdot\text{m}^{-3}$.

Tab. 6 The middle distillates characterization

Sample (t/WHSV)	Kin. viscosity 40 °C ($\text{mm}^2\cdot\text{s}^{-1}$)	Density 15 °C ($\text{kg}\cdot\text{m}^{-3}$)
EN 590	2.0-4.5	820-845
305/1	2.70	778.3
310/1	2.78	778.2
315/1	2.51	- ^a
320/1	2.17	773.2
325/1	- ^a	- ^a
330/1	1.81	767.2
340/1	1.74	768.1
330/2	2.31	775.1
340/2	1.79	766.0
350/2	- ^a	- ^a
360/2	- ^a	- ^a
330/4	2.57	778.0
340/4	2.57	777.6
350/4	2.31	- ^a
360/4	1.83	768.5
370/4	- ^a	- ^a

^{a)} was not measured due to the insufficient amount of sample or poor low-temperature properties

The kinematic viscosity of the middle distillates obtained at the lower reaction temperatures, i.e., up to 320/1, 330/2 and 350/4, met the EN 590 requirements for moderate climate diesel, all the other middle distillates met the viscosity requirements for arctic diesel. Although the obtained middle distillates did not comply with the

current EN 590 standard, they could be used as components into diesel fuels in an appropriate concentration.

4. Conclusion

The hydrocracking of the FT wax was carried out in a fixed-bed reactor at the reaction temperatures of 305-370 °C, a pressure of 8 MPa and a WHSV of 1; 2, and 4 h⁻¹.

The yield of the gaseous products increased with the reaction temperature and decreased with the WHSV. They were primarily composed of C₃-C₅ n-alkanes and C₄-C₅ isoalkanes and could be, therefore, used for LPG production or as feedstock for steam cracking.

All the obtained liquid products were mainly composed of a naphtha fraction. The content of the aromatics, benzene and olefins in all the naphtha fractions was below the particular limits of the EN 228 specification. The DVPE of the naphtha fractions obtained from the products formed at higher hydrocracking temperatures met the EN 228 specification requirements. Many naphtha fractions also complied with the specification regarding the parameters of the distillation test. Nevertheless, the densities of all the naphtha fractions ranged between 651.5 and 701.7 kg·m⁻³ which is much lower than the lower specification limit of 720 kg·m⁻³. The neat or processed (isomerization or reforming) naphtha fraction obtained from the FT wax hydrocracking could be used as a component for gasoline production, for the production of a low-sulfur and low-aromatic paraffinic solvent or as a feedstock for steam cracking.

The middle distillates only formed a minority product with a maximum yield of 12 wt.%. All the obtained middle distillates complied with the EN 590 specification regarding the content of the polycyclic aromatic hydrocarbons. Most of the samples met requirements for kinematic viscosity. The densities of all the samples were significantly below the limitation.

It can be concluded that the hydrocracking of the FT wax required much milder reaction conditions when compared to the hydrocracking of petroleum vacuum distillates. On the other hand, the yields of the middle distillates were lower than was observed by other researchers. Further experiments will, therefore, be focused on the FT wax hydrocracking with different types of catalysts and with the aim to increase the yield of the middle distillates, especially the kerosene fraction.

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