Pyrolýza a přímé zkapalňování uhlí, vliv přídavků malténů na hydrokrakování vakuového plynového oleje

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Abstrakt

Malténová frakce z pyrolýzního oleje z hnědého uhlí nebo produkt přímého zkapalňování uhlí s bodem varu nad 220 °C byly smíseny s vakuovým plynovým olejem pro následné hydrokrakování. Cílem práce bylo zjistit, jakým způsobem ovlivní přídavek produktů z uhlí do vakuového plynového oleje vlastnosti finálních produktů (ve srovnání s čistým vakuovým plynovým olejem). Byl použit katalyzátor na bázi nikl-wolframu na siliko-alumině. Testy byly provedeny v laboratorní jednotce (rychlý screening) a na kontinuální pilotní jednotce. Při testech na laboratorní jednotce byly produkty analyzovány simulovanou destilací. Při pilotních testech byly plynné a kapalné produkty analyzovány simulovanou destilací, plynovou chromatografií GC-FID/TCD a GC-MS. Přídavek produktů z uhlí proces hydrokrakování výrazně neovlivnil.

Pyrolysis and direct coal liquefaction - maltenes addition effect on the vacuum gas oil hydrocracking

The maltenes fraction of coal tar pyrolysis or direct coal liquefaction product with a boiling range higher than 220 °C was mixed with vacuum gas oil for hydrocracking. The aim of the work was to study how an addition of coal derived compounds addition to vacuum gas oil affects the final product characteristics (comparing the tests using pure vacuum gas oil). Nickel tungsten supported on silico-alumina catalysts were used. Tests were performed in bench-scale (fast screening) and pilot-scale continuous flow reactors. For the fast screening, the feedstock and liquid products were analysed by simulated distillation. For longer-term tests, gases and liquids were analysed by simulated distillation, GC-FID/TCD, and GC/MS. The addition of coal-derived liquids did not affect the overall hydrocracking process.

Die Pyrolyse und direkte Kohleverflüssigung, Wirkung des Zusatzes von Maltenen auf das Hydrokracken des Vakuumgasöls

Die Maltenfraktion aus dem Pyrolyseöl aus Braunkohle oder das Produkt der direkten Kohleverflüssigung mit dem Siedepunkt über 220 °C wurden mit dem Vakuumgasöl für das anschließende Hydrokracken gemischt. Ziel der Arbeit war festzustellen, auf welcher Weise der Zusatz von Kohleprodukten in Vakuumgasöl Eigenschaften der Finalprodukte beeinflusst (im Vergleich mit reinem Vakuumgasöl). Es war ein Katalysator auf Basis Nickel-Wolfram an das Siliko-aluminium eingesetzt. Die Tests wurden in der Laboreinheit (schnelles Screening) und auf einer kontinuierlichen Piloteinheit vorgenommen. Bei den Tests in der Laboreinheit wurden die Produkte durch simulierte Destillation analysiert. Bei den Pilot Tests wurden die gasförmigen und flüssigen Produkte durch simulierte Destillation, Gaschromatografie GC-FID/TCD und GC-MS analysiert. Der Zusatz der Kohleprodukte hat den Prozess Hydrokracken erheblich nicht beeinflusst.

Klíčová slova: ropa, uhlí, zkapalňování, dehet, vakuový plynový olej, koprocesing.
Keywords: crude oil, coal, liquefaction, tar, vacuum gas oil, co-processing.

1 Introduction

Coal liquefaction is a well-known process that was developed since the 1930s. It was used in a big scale by the German army for producing synthetic fuels [1]. Coal liquefaction is done by different methods: (i) indirect coal liquefaction which is carried out by the gasification of the coal to produce syngas (the syngas will be transformed to hydrocarbons or other oxygenated compounds), (ii) direct coal liquefaction in which the coal is transformed into liquids with or without the use of catalysts or solvents [2]. Thus, a source of synthetic crude could be used to be mixed with fossil petroleum for co-processing. Especially, taking into account that European coal resources are present in much higher amounts compared to petroleum resources. Although renewable feeds are being used in refinery, the European Union is still a net petroleum importer and according to EU's Oil Stocks Directive (2009/119/EC), each country must maintain emergency stocks of petroleum. Besides, some of the available petroleum oil resources are located in unstable territories and the demand for petroleum is continuously increasing [3]. So, the use of coal sources can be a temporary solution while the environment friendly use of renewable sources is gradually increasing up to 100%. The synthetic crude oil can be distilled in various fractions and these fractions can be co-processed with its respective fossil petroleum boiling range fractions. The hydrocracking of vacuum gas oil is performed to produce lighter products such as gasoline, kerosene or diesel. The middle distillates are the most suitable products [4]. The hydrocracking of pure vacuum gas oil is a well-known process [5-7]. The coprocessing of vacuum gas oil with other

Table 1: Coal tar elemental C, H, S % analyses and simulated distillation (SIMDIS) results.

S, wt%	0.61	**SIMDIS °C**	**IBP**	187	**30 %**	294	**80 %**	413
C, wt%	55.8		**10 %**	245	**50 %**	338	**90 %**	453
H, wt%	8.19		**20 %**	270	**70 %**	381	**FBP**	584

compounds is also known [8-11]. A mixture of Fischer–Tropsch waxes (10 wt%) and vacuum gasoil was tested obtaining higher hydrocracking conversions [8]. Canola, sunflower or waste cooking oil were also used mixed with vacuum gas oil [9-11].

Although the use of coal liquids for the hydrocracking coprocessing with heavy petroleum oils is a known procedure [12], the use of coal tar and direct coal liquefaction products for the coprocessing (hydrocracking) with vacuum gas oil was only studied in bench and pilot scales during the project DIRPRIMCOAL (Direct Primary Coal Liquefaction via an Innovative Co-processing Approach with Waste and Petroleum Feedstocks). The results are exposed in this work. This work aimed to evaluate the coal tar and direct coal liquefaction products addition effect on the hydrocracking of vacuum gas oil using a commercial catalyst (reference material) and laboratory synthesized catalysts. The heavy fraction (maltenes), containing a boiling range of >220 °C, was selected to be tested with vacuum gas oil. The maltenes fraction was selected to avoid the lack of solubility of the polar compounds and water with vacuum gas oil which could result in many problems in the reactor such as blocking the pipes and valves.

2 Experimental

2.1 Raw materials

The research catalysts and the standard reference commercial material used for this work were based in NiW supported on silica-alumina. Catalysts´ details are confidential.

The vacuum gas oil was obtained directly from the refinery (300 – 500 °C boiling range fraction. It contained 84.9, 9.94, 1.92 wt% of C, H, S respectively and 847 mg.kg^{-1} of N. The vacuum gas oil density at 50 °C was 875 kg.m^{-3}.

Coal tar was obtained using a previously published method [13-15]. The test for the pyrolysis of the brown coal was performed in a batch reactor [13, 14] from room temperature to 650 °C (5 °C min^{-1}) maintaining the temperature at 650 °C during 3 hours. The organic coal tar was distilled up to 220 °C removing the lighter fraction and keeping the heav

Table 2: GC/MS analysis of pure direct coal liquefaction product (> 220 °C boiling range fraction).

Identified Compound	Rel. % by peak area
decaline	0.15
Me-Indane	0.18
decaline isomer	0.14
tetralin	15.83
naphthalene	10.31
2-Me naphthalene	2.34
1-Me naphthalene	8.78
C2 naphthalenes	4.67
C3 naphthalenes	3.70
C4 naphthalenes	1.95
phenanthrene	0.37
Sum Identified Comp.	48.42
Sample up to phenanthrene	76.90
Whole sample	100.00

Table 3: GC/MS analysis of pure coal tar product (> 220 °C boiling range fraction).

Compound	Peak GC Area %
alkanes	53.4
alkylbenzenes	19.2
indanes	12.2
naphthalenes	2.5
biphenyles	0.7
fluorenes	1.5
phenanthrenes	2.5
fluoranthenes	1.8
Policyclic Aromatic Hydrocarbons (PAH)	4.2
C1-4 BT (benzothiophenes)	0.6
DBT (dibenzothiophenes)	1.5

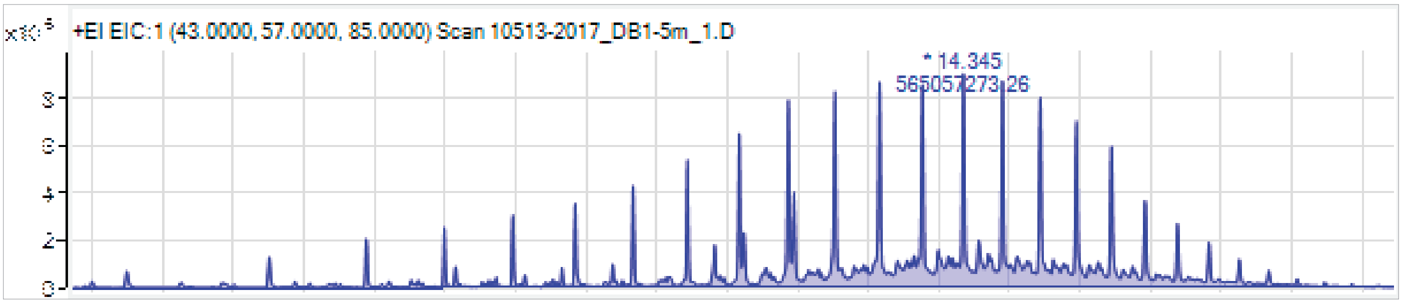

Figure 1: GC-MS chromatogram. Coal tar with a boiling range of >220 °C, compounds from C9 to C33 with the maximum located at C21 and C22 compounds.

one. The maltenes fraction was obtained by solubilizing the heavy fraction (boiling range of >220 °C) in n heptane. Then, the heptane was removed by distillation obtaining the raw material (Table 1, 3). Then, it was mixed with vacuum gas oil in a weight ratio of 10:90 of coal tar:vacuum gas oil.

Direct liquefaction raw material (boiling range of >220 °C, Table 2) was obtained by the mixture of the products from previous tests described in a previous publication [3]. The composition of coal tar with boiling range >220 °C is given in Table 3 and Figure 1.

2.2 Tests

2.2.1 Screening tests

For the screening tests, a continuous flow reactor with twenty points for temperature measurement sensors was used. The length of the reactor was 1 058 mm (top position = 0 mm and bottom position = 1 058 mm) with the measurement points between 253 and 815 mm. The catalyst was mixed with SiC (1:1 by volume). As a standard procedure, some quartz wool was located at the bottom of the reactor and then 10 cm^3 of pure SiC (0.5 mm particle diameter) was added. As a second step, the catalyst + SiC mixture (1:1 volume ratio) was added. Finally, the rest of the reactor was filled with pure SiC.

First, the catalysts were activated using a solution of 2 %wt. dimethyldisulphide (DMDS) in atmospheric gas oil (AGO) at 350 °C. Then, research and commercial reference catalysts were tested at 420 °C and 18 MPa hydrogen pressure. The test conditions for the research catalysts and commercial catalyst are presented in Table 4. The different amounts of catalyst were selected taking into account the available amount of catalyst and the reactor conditions (maximum and minimum limit of gases and liquids mass flow).

2.2.2 Pilot test

A continuous flow reactor (inner diameter of 40 mm, a pipe (thermal probe) diameter of 10 mm and a reactor length of 1 200 mm) was used. The input previous to the pump was heated at 60 °C to avoid solid feedstock formation. The output of the reactor (for gases and liquids sampling) was maintained at 24 °C. The reactor bottom was filled with quartz wool. Then, pure SiC was added over the quartz wool up to the catalyst bed which was filled with 100 g of the catalyst NiW 7p (the most active catalyst selected was NiW-7 and scaled up to NiW-7p) mixed with SiC in a 1:1 volume ratio. The rest of the reactor was filled with pure SiC. The catalyst was sulfidized-activated at 350 °C during 72 hours with a feed rate of 250 g/h (Atmospheric Gas Oil + Dimethyl Disulphide 2 wt%) and a pressure of 4.5 MPa. Then, the reactor was flushed with N_2 and heated to reaction conditions (375 °C, 18 MPa bar [H_2], WHSV = 1 h^{-1}, 110 Nl/h [H_2]).

2.3 Analyses

2.3.1 Screening tests products analyses

The simulated distillation (SIMDIS) was performed by gas chromatography following the ASTM D2887. Gases were analysed by the method ‟Refinery Gas Analysis RGA" using GC-FID/TCD 7890A Agilent (Agilent Technologies), configured to analyse up to C6 hydrocarbons including H2S and carbonyl sulphides (COS) [3].

GC-MS of the n-heptane solubles from the coal tar was carried out using a Trace Ultra MS: DSQ II (Thermo Scientific) system. GC column: HP5ms (Agilent Technologies). Compound identification: NIST 14 MS Database + GC RI (Retention Index) NIST database [3].

2.3.2 Pilot test products analyses

Liquid samples were collected at 24 °C and gases collected using inert foil gas sampling bags were analysed as in the previous reactor.

Complete liquid samples were analysed using simulated distillation (also C, H, N, S% elemental analysis was carried out for selected samples). Then, collected samples were mixed in homogeneous intervals of time and distilled in three fractions, < 180 °C, 180-230 °C, 230-360 °C and > 360 °C. Finally, they were analysed by refractive index, GC-FID (PIONA), elemental C, H, N, S % analysis, density (15 °C), ASTM D1500 colour, cloud point, aniline point, pour point, aromatics content by HPLC. Gas products were analysed by GC-FID/TCD – RGA (Refinery Gas Analysis).

Table 4: Hydrocracking screening fast tests carried out (420 °C, 18 MPabar)

Test	Catalyst	Catalyst [g]	VGO flow rate [g/h]	WSHV [h^{-1}]	Hydrogen flow rate [dm^3/h]
I	Reference catalyst	42.65	37	0.87	50
II	NiW-1	42.65	37	0.87	50
III	NiW-2	42.65	38	0.89	50
IV	NiW-3	26	23	0.88	50
V	Reference catalyst	26	80	3.08	110
VI	NiW-4	26	80	3.08	110
VII	NiW-5	26	80	3.08	110
VIII	NiW-6	26	80	3.08	110
IX	NiW-7	26	80	3.08	110

Table 5: Summary of the SimDis results from screening the research catalysts.

Test	Test catalyst used	ABO[1] %wt	O[2] %wt	AAO[3] %wt	TOS[4] [h]	ADi-ADf[5] %wt
I	Reference catalyst	58.5	--	--	--	63-54
II	NiW-1	15	14	17	24 to 36	25-17
III	NiW-2	16	15	18	18 to 30	16-17
IV	NiW-3	40	19	18	18 to 30	38-19
V	Reference catalyst	26	--	--	--	30-22
VI	NiW-4	15	29	34	6 to 15	15-34
VII	NiW-5	11	12	12	6 to 15	11-12
VIII	NiW-6	17	11	11	6 to 15	17-11
IX	NiW-7	100	100	100	6 to 15	31-39

[1] (ABO) Average amount distilled up to 300 °C from the product collected before feeding n-heptane soluble pyrolysis oil (10 %wt. in VGO). In this step, only pure VGO was used as feedstock.

[2] (O) Average amount distilled up to 300 °C from the product obtained with feeding the n-heptane soluble pyrolysis oil (10 %wt. in VGO).

[3] (AAO) Average amount distilled up to 300 °C from the product obtained after feeding of n-heptane soluble pyrolysis oil added (10 %wt. in VGO). In this step, only pure VGO was used as feedstock.

[4] (TOS) Time on stream at which the n-heptane soluble pyrolysis oil was added.

[5] (ADi) Amount distilled of the first collected sample amount (average liquid collected after the first hour of reaction at 1 °C in the output of the reactor) – (ADf) amount distilled of the last collected sample (average liquid collected one hour before the end of the test, at 1 °C in the output of the reactor). These amounts are related to the product amount distilled up to 300 °C from the complete product.

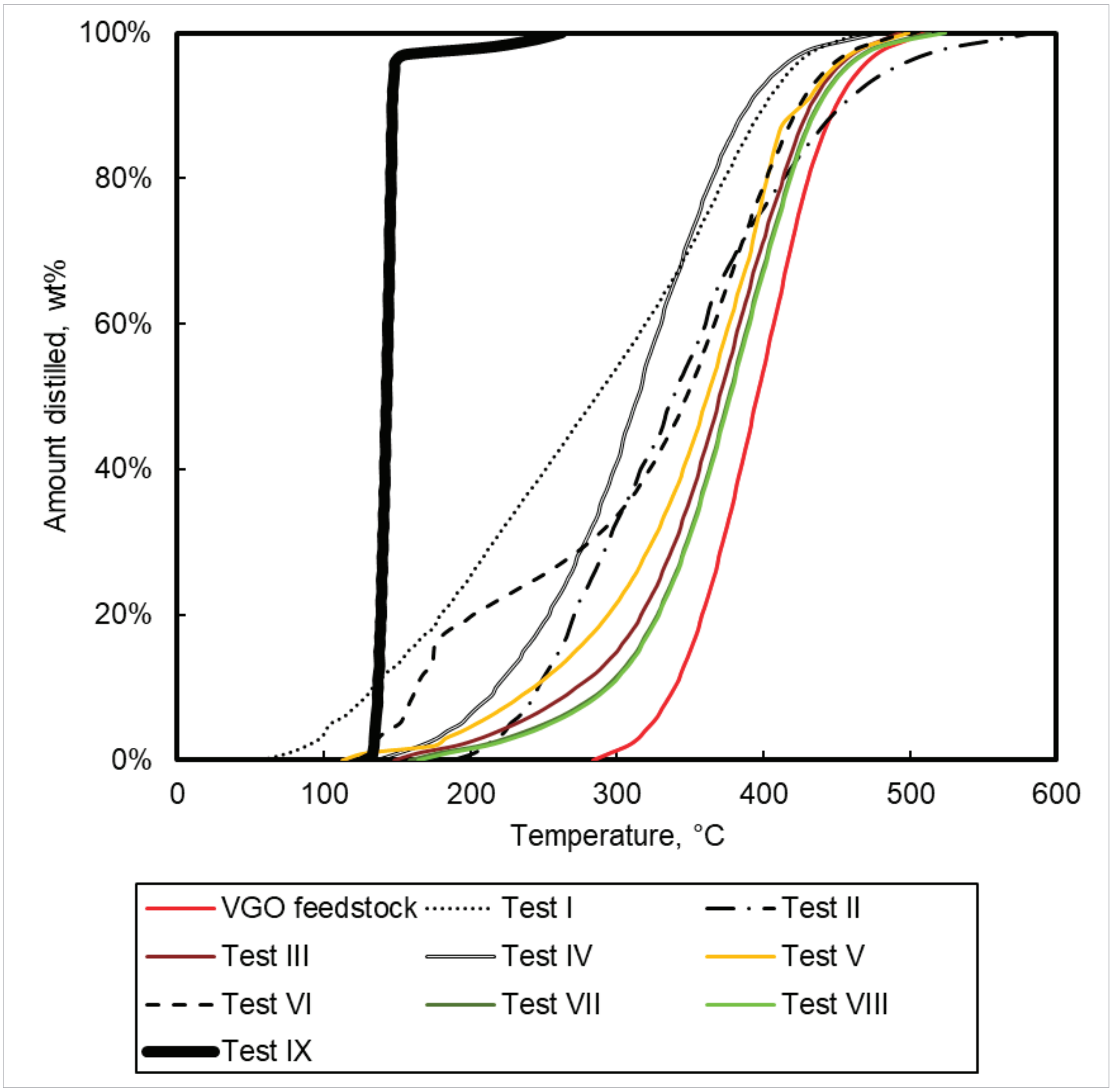

Figure 2: Representative SimDis analyses of tests products and vacuum gas oil (VGO) feedstock.

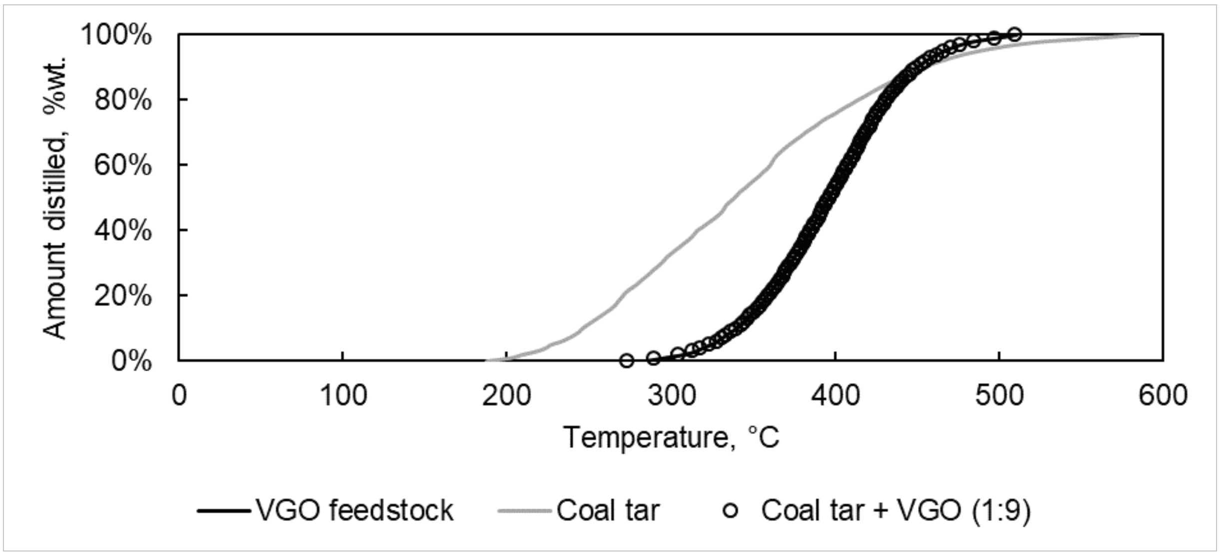

Figure 3: Coal tar, VGO and Coal tar + VGO (1:9 weight ratio) feedstock SIMDIS.

3 Results and discussion

3.1 Screening tests

For a fast evaluation, the SIMDIS analysis of the products was used to determine the amount of hydrocracked products with a boiling range up to 300 °C (Table 5, Figure 2). The reference catalyst test led to the lightest compounds as shown in Figure 2. However, test IX presented only light products (100 wt% with boiling points lower than 300 °C).

The VGO feedstock did not contain any distillable compounds boiling below 300 °C. The feedstock, coal tar and the mixture 10:90 wt:wt of coal tar and vacuum gas oil SIMDIS are represented in Figure 2.

NiW-4 and NiW-7 catalysts (tests VI and IX) led to the highest conversion for vacuum gas oil hydrocracking (adding coal tar and after adding coal tar using only pure vacuum gas oil) compared to the reference commercial catalysts (Table 5). NiW-3 presented an initial high activity but the vacuum gas oil conversion decreased when the coal tar n heptane solubles were added (the coal tar was added at a time on stream of 18 h). Catalyst NiW-7 presented too high activity with a high yield to gases C1-C4 so it was selected temporarily to continue with the tests to study the catalytic activity at a lower temperature (Figure 3). In this case, the products were analysed by density, simulated distillation and, mass balance to have a fast methodology to find the most active catalyst. The yield to products with a boiling range lower than 300 °C activity was not affected by

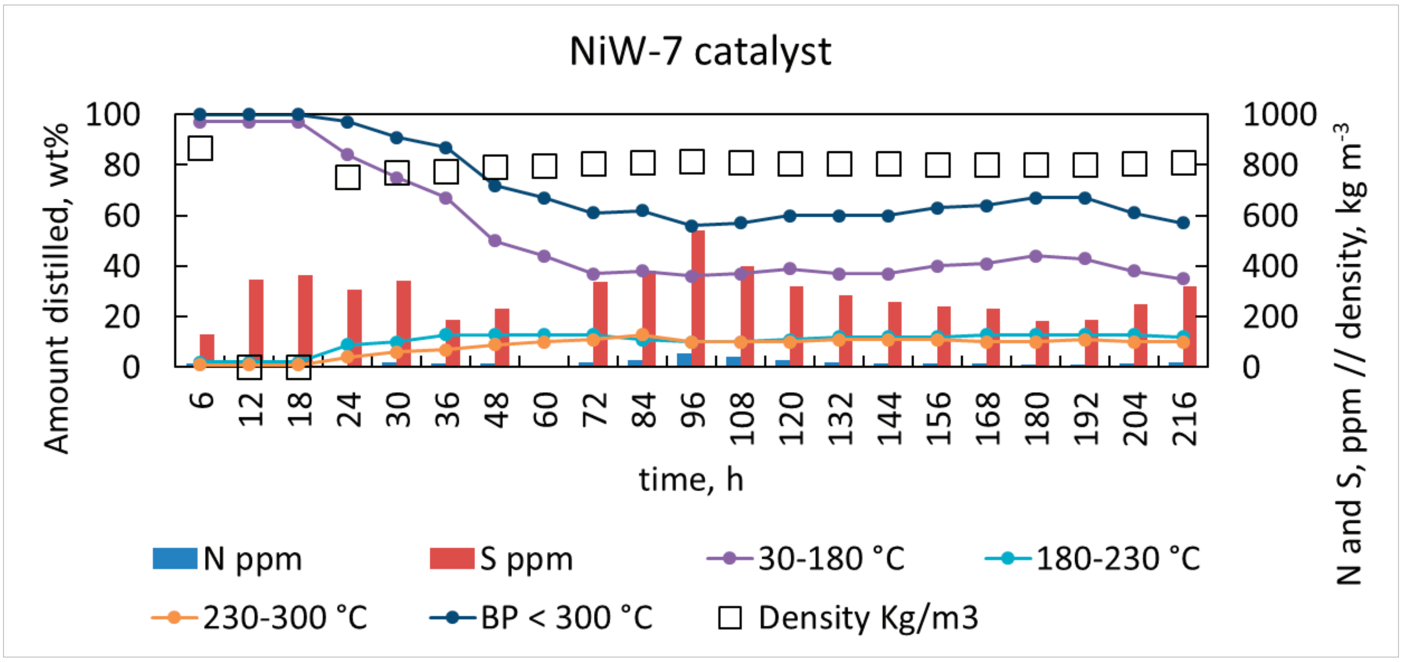

Figure 4: Test carried out in the reactor for the screening tests during a time of 216 hours. WHSV = 3 h^{-1}, 420 °C. Starting the reaction at 400 °C. Boiling range of the products at 30-180 °C (violet colour), 180-230 °C (blue colour), 230-300 °C (orange colour) and <300 °C (all products under b. r. of 300 °C, dark blue colour). N ppm and S ppm are the amounts of nitrogen and sulphur (mg kg^{-1}). Direct coal liquefaction product was added (10 %wt) to the vacuum gas oil from 48 h to 120 h.

the addition of coal tar maltenes. So, the fresh activated-sulfided catalyst NiW-7 was tested (Figs. 3-5).

In Figure 3, the most active catalyst presented a continuous stable activity after 72 hours of reaction. The reaction started at 400 °C, during the first 24 hours due to high catalytic activity and exothermic reaction obtaining a high yield to gases C1-C4. After 24 h, the conversion to gases was high with a yield of 80 wt% to liquids (20 wt% to gases). Direct coal liquefaction maltene fraction product was added from 48 to 120 h (10 wt% of direct coal liquefaction product to 90 wt% of vacuum gas oil). The addition of the mixture of vacuum gas oil and direct coal liquefaction maltene product did not affect dramatically to the catalytic activity. However, the yield to gases was still too high so it had to be changed so possibly decreasing the temperature. The next step was to use the pilot unit (Figure 4) and use less temperature and also less WHSV (weight hourly space velocity) to obtain better yields to liquids during the hydrocracking reaction.

3.2 Pilot tests

Because a high yield to gases was found. The test for selecting the temperature in the pilot unit led to know the mass balance to liquids and gases (Figure 4). The optimum temperature

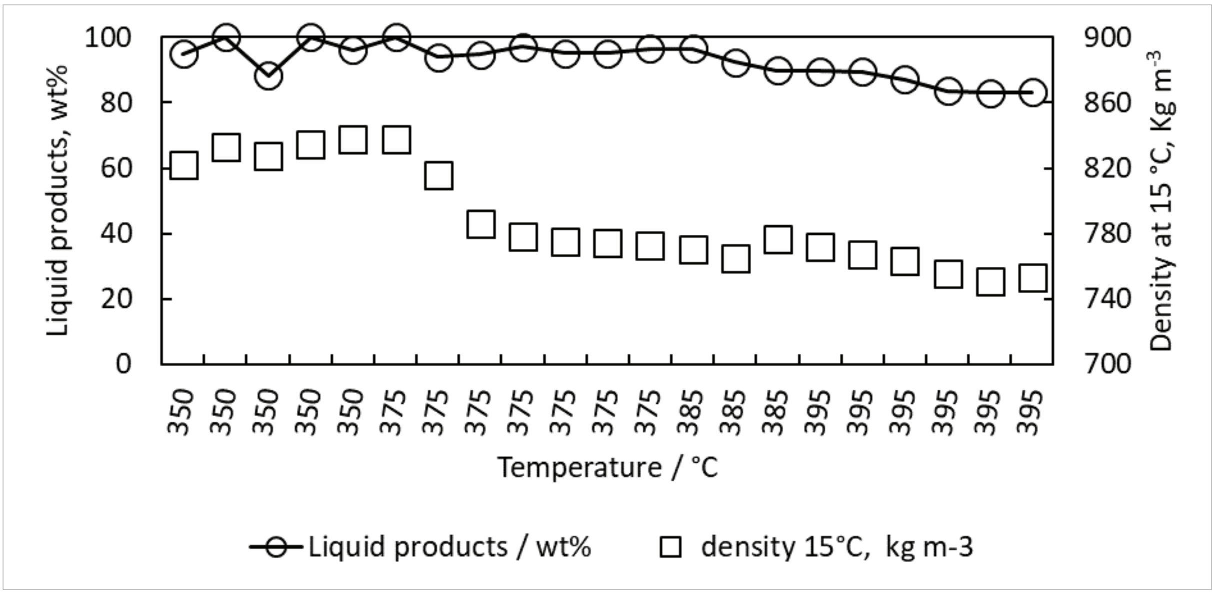

Figure 5: Test using NiW-7 catalyst at 350, 375, 385 and 395 °C at WHSV = 1 h-1 and 100 g of catalyst in the pilot unit for selecting the best temperature.

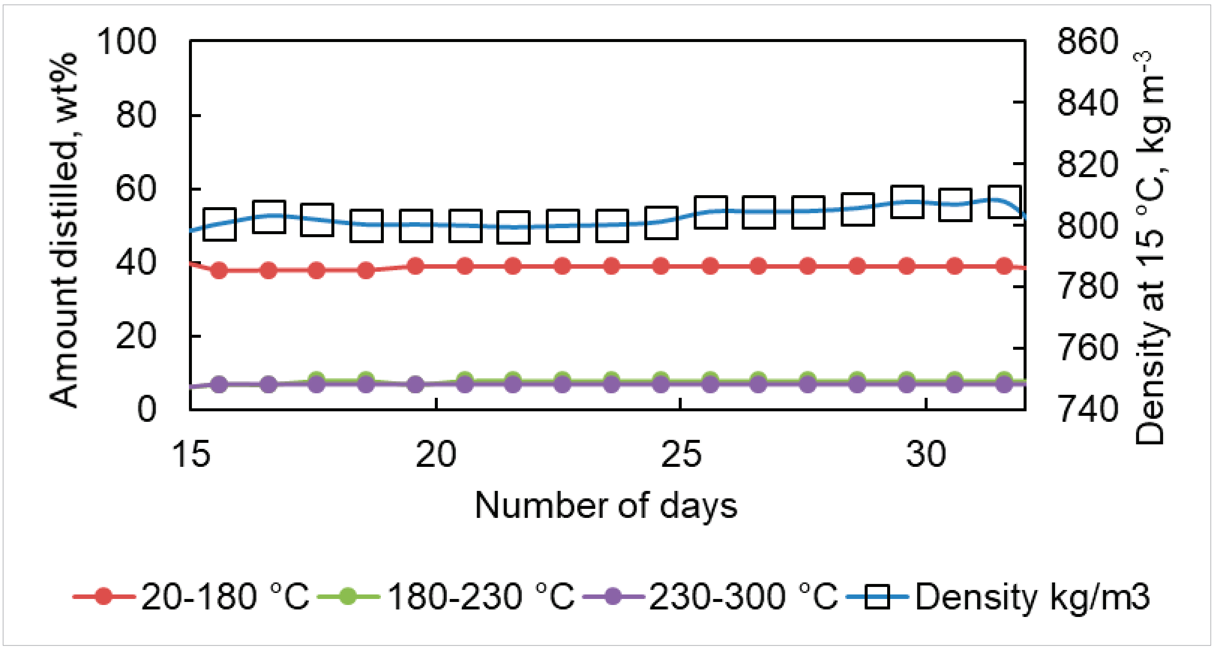

Figure 6: Test using NiW-7 catalyst at 375 °C at WHSV = 1 h^{-1} and 100 g of catalyst in the pilot unit. Boiling range of the products at 20-180 °C (violet colour), 180-230 °C (blue colour) and 230-300 °C (orange colour). Direct coal liquefaction product was added (2 %wt) to the vacuum gas oil.

Table 6: Representative Analysis of liquid products mixture (from 19th day to 29th day). In some cases not enough amount of sample was found and some analyses could not be done.

Temperature [°C]	<180	180-230	230-360	>360
Amount distilled, wt%	39.00	2.20	18.40	39.60
N, mg/Kg	less than 0.5	2.12	49.5	129
S, mg/Kg	173	60	729	1385
C, wt%	84.9	not enough	87.1	86.4
H, wt%	14.8	not enough	13	13.6
Density [15°C] kg/m^3	738	841	861	846 (50°C)
Refractive Index	1.41	1.46	1.48	1.47
PIONA, Calc. Octane Number	69	10	n	n
ASTM color D1500	less than 0.5	not enough	0.4	5.5
Cloud Point, °C	n	not enough	-3	n
Aniline point, °C	n	not enough	69	n
HPLC, Monoaromatics %	n	34.6	34.7	n
HPLC, Diaromatics %	n	1.3	2	n
HPLC, Polyaromatics %	n	0.1	0.5	n
Pour Point, °C %	n	not enough	-11	28

was 375 °C with a 95-97 wt% of liquids yield. Thus, 375 °C was selected for continuing with the pilot unit for the long-term test.

The long-term test was carried out up to 77 days. The conversion was variating from 42-46 wt% (up to 50 days of stable activity) to 28 wt% (day number 77). This conversion was related to products with a boiling range lower than 300 °C. The direct coal liquefaction product was added in the amount of 2 wt% to have enough feed mixture (98 wt% of vacuum gas oil and 2 wt% of direct coal liquefaction maltene fraction) during all the test. During the test, the addition of direct coal liquefaction maltenes did not suppose any apparent change to the activity of the reaction.

The highlights of the work were:

- Hydrocracking tests performed in bench and pilot scale reactors. The feedstock weres) coal tar and vacuum gas oil maltenes.
- Coal tar and direct coal liquefaction maltenes (tests carried out with a maximum of 10 wt%) with a boiling range of >220 °C can be used for the vacuum gas oil hydrocracking coprocessing.

The mixture of the liquid products from the 19th to the 29th day was analysed to know the average properties of these liquids at the boiling ranges of <180, 180-230, 230-360 and >360 °C.

The gaseous products were also analysed (3-5 wt% yield to gases) obtaining mainly methane (Figure 6).The amount of methane was decreasing with the time on stream but the C3-C5 gaseous products contents increased.

4 Concluding Remarks

Several tests of hydrocracking of vacuum gas oil mixed with maltenes from the vacuum gas oil fraction of coal tar and direct

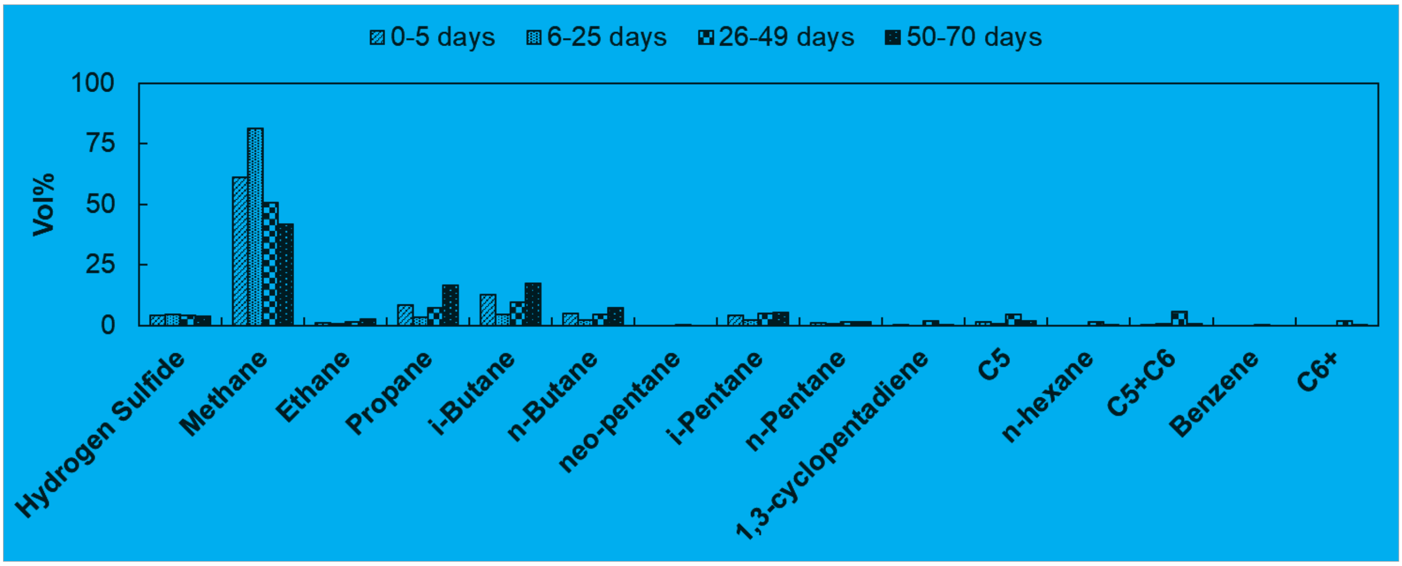

Figure 7: RGA analyses for the gaseous phase obtained during the pilot unit tests.

coal liquefaction products were performed using NiW supported catalysts. The addition of maltenes derived from coal tar or liquid from direct coal liquefaction did not affect the overall activity of the process. Tests were performed in bench-scale (fast screening) and pilot-scale continuous flow reactors. A high activity was found at a low temperature of reaction (375 °C) for one of the research catalysts obtaining a yield of approximate 50 wt% to products with a boiling point lower than 300 °C.

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