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TESTING OF HYDROTREATED VEGETABLE OIL AS BIOCOMPONENT IN DIESEL FUEL

Abstract

INA as a motor fuels distributor has an obligation to put on the Croatian market a mandatory share of biofuels in the transport energy mix according to the Act on Market of Biofuels for Road Transport. INA started to use fatty acid methyl esters (FAME) as biocomponent for diesel fuel blending in 2011. The significant increase of biofuels energy share, in next few years requires the introduction of a new type of biocomponents that are suitable for processing in INA refineries. Hydrotreated vegetable oil (HVO) is one type of renewable synthetic fuels, the so called drop-in fuel. HVO is used as a high quality diesel blending component, or as standard diesel fuel, very similar to the gas to liquid (GTL) and biomass to liquid (BTL) fuels. The use of HVO enables better vehicle performance and emission reduction compared with conventional diesel. It is practically aromatics and sulphur-free and has high cetane number. Therefore, HVO shows superior quality compared with convention diesel and FAME. An additional interesting property is its very low density compared to common diesel fuel because INA is occasionally processing the naphthenic West African grades with gas oils of a very high density.

The possibility of blending of the HVO component with diesel fuel and iwith the mixture of diesel fuel and FAME was tested in order to meet HRN EN 590 standard for diesel fuel and to examine the impact of HVO addition on important diesel's properties. A maximum amount of 18 vol. % of HVO was added in tested diesel fuel in order to meet EN 590 standard regarding density value. Density decrease and a significant cetane number increase were noticed in blends. In the tested blend of HVO and diesel fuel with FAME the same density like in diesel fuel and cetane number increase like in the HVO and diesel fuel blend were noticed. Distillation range did not change significantly in both types of blends. The added HVO has not significantly influenced the improvement of low temperature properties (cloud point (CP), cold filter plugging point (CFPP)) of all tested blends. The slightly better response of cold flow improver (CFI) additive and achieved better operability was noticed in blend with HVO, diesel fuel and FAME.

Considering the results obtained in INA, blending of HVO with diesel fuel and FAME is more acceptable because of achieving better operability and high cetane number.

The results obtained for two blends analyzed in laboratory are compared with results calculated by commercial software which is used for estimation of motor fuel properties. A relatively good agreement was achieved regarding the main estimated properties expect the cetane number. It can be concluded that software could be successfully used for predicting the behavior of blend of HVO with diesel fuel or with diesel fuel and FAME mixture, apart from cetane number. It can be used for obtaining fast results, selection of targeted blending recipe and the laboratory analysis. The testing of HVO blending with diesel will be continued in order to optimize the recipe and to examine the influence on low temperature properties.

Key words: biocomponent, hydrotreated vegetable oil (HVO), drop-in fuel

1. Introduction

INA started blending FAME as biocomponent in diesel fuel. The share of energy from biofuels and biocomponents is defined by National action plan and it increases every year. In 2016, the introduction of biocomponent in motor gasoline is planned in order to achieve the planned share of biocomponent as a renewable energy sources. INA is looking for a new biocomponent to be used in order to meet greenhouse gas emission requirements according to Fuel Quality Directive 98/70/EC and Directive 2009/28/EC (RED). Refining and Marketing Development Sector has investigated HVO as a potential biocomponent for blending with diesel because of its good physical, chemical and blending properties. HVO is a high quality paraffinic diesel fuel produced from vegetable oils and/or animal fats by hydrotreating and isomerization. Companies Axens, IFP, Honeywell, UOP, Neste Oil, Syntroleum and UPM have developed their own technologies for HVO production [1,2]. HVO and other synthetic fuels (GTL, CTL or BTL diesel) are also known as *drop-in fuels* because they can be used as blending components in diesel fuel or as a pure diesel. If HVO is used as fuel on its own, significant reduction of NO_x and particulate emission is noticed. This makes HVO suitable as diesel fuel for urban traffic e.g. city buses.

HVO is accepted by Fuel Quality Directive, Annex II for diesel fuels and due to its advantages it is commonly used in some EU countries. HVO also meets requirements related to the conventional diesel (EN 590, ASTM D975 or D396, Worldwide Fuel Charter Category 4) except for the low density in some standards [3].

Density of HVO is lower in comparison with diesel fuel due to paraffinic nature and low final boiling point (FBP). Density of diesel fuel is traditionally an important characteristic which has a positive effect on the engine power output and volumetric fuel composition. If density reduces, the heating value per volume decreases as a function of density. The HVO behavior differs and has the higher energy per mass that compensates partly the lower density effect.

The HVO share in diesel fuel is not limited in comparison to max 7 vol. % of FAME according to the Fuel Quality Directive and EN 590 standard. The only potential limit regarding blending is the low density of HVO relative to the density of blended diesel fuel. Usually about 20 to 30 vol. % HVO is added depending on diesel density. ASTM D975 method [4] has no density limit, so HVO can blend in all ratios without blending wall or labeling at fuel stations.

HVO is aromatics and sulphur free that results with low tail pipe emissions. Viscosity of all drop-in fuels is close to that of traditional diesel fuels, as opposed to FAME, which displays as high as almost double viscosity.

The HVO are produced according to local requirements whereat their low temperature properties vary significantly, in the range from -5 to -30 °C. They have pretty high cetane number that make them desirable component for blending of premium diesel fuel. HVO energy content per mass is higher than for fossil diesel due to the fact that HVO's hydrogen content is about 15.2 wt. % compared to 13.5 wt. % for diesel fuel. HVO has a heating value of 44.1 MJ/kg, while FAME has 37.3 MJ/kg (difference in comparison to DF: HFO -5 %, FAME -9 %).

The distillation range of HVO is also inside the conventional diesel fuel range and below the FAME, which shows a quite high-boiling range. The HVO does not have a negative effect on FBP area of diesel fuel as FAME.

HVO is characterized by good stability. Method for determination of stability of FAME or of diesel fuel with 2 to 7 vol. % of FAME (EN 15751 “Rancimat” method) is not suitable for HVO because of its specific composition. HVO is fully paraffinic and its solubility is low in conventional diesel which contains 15 to 30 % of total aromatics. The existing impurities from diesel or FAME may precipitate when FAME is mixed with low aromatic or aromatic-free fuel and with low density components. Therefore, it is recommended to start blending of diesel fuel with HVO, and subsequently FAME may be added to this blend. Also, the storage stability problems are diminished when compared to blending with FAME due to the decreasing possibility of water separation, which causes micro-biology contamination. Furthermore, HVO does not cause dilution or deterioration of engine oil.

Automotive manufacturers prefer HVO over FAME since HVO reduces NO_x emissions, particulate, CO, PAH and GHG emissions. Thus, when HVO is used as a blending component in diesel fuel, NO_x and particulate emissions reduce linearly as function of blending ratio.

The determination of the HVO share as in fuel biocontent can possibly be a problem. However, it can be determined by two methods based on ^{14}C: Liquid Scintillation Counting (LSC) and Accelerated Mass Spectrometry (AMS). These methods are available in some commercial laboratories, but due to the time consumption and other disadvantages this is not practical for everyday use.

2. Experimental part, results and discussion

The possibility of blending HVO with diesel fuel and with diesel fuel and FAME was tested and discussed.

2.1 Samples

The analyses were performed on the following samples: diesel fuel from hydrocracking unit originated from low sulphur crude oil, winter cut, Croatian originated FAME and commercial HVO. Physical and chemical characteristics, hydrocarbon composition, *n*-paraffins content and distribution in components and blends were analyzed. Properties of blends were estimated with the assistance of commercial software, while cetane number was determined by cetane engine method (HRN EN ISO 5165 method).

2.2 Properties of blending components and blends

Results of analyses of physical and chemical properties of blending components are given in Table 1 and Table 2. Diesel fuel quality is in accordance with HRN EN 590 standard [5].

The properties of HVO which could have impact on the diesel quality were analyzed. HVO density is below minimum value required for the density of diesel fuel. In view of that the maximal amount of HVO in diesel fuel was determined.

Cetane number of HVO is very high and it varies between 75 and 95 due to its nature, i.e. content of n- and iso-paraffins. In the case of using HVO as blending component, cetane number increases linearly. If HVO is used directly as a pure diesel fuel, it can be proclaimed as diesel fuel of premium quality. Cetane number of pure HVO was determined by standard test method for determination of derived cetane number of diesel fuel and gas oils (ASTM D 6778-14a).

A typical distillation range of HVO is within that of conventional diesel fuel, as opposed to FAME, which contains significantly heavier compounds and has wider distillation range and causes the increase of the blend FBP.

Pure HVO has excellent low temperature properties because of the possibility to change the isomerization process severity [6,7]. But its narrow distillation range and narrow carbon chain distribution (C_{15}-C_{18}) of paraffinic hydrocarbons are very challenging regarding the performance of the cold flow additive.

The key characteristics for monitoring of FAME quality were analyzed: density, water and sediment content, oxidation stability, viscosity and cold filter plugging point (CFPP). All properties are in accordance with the requirements of the HRN EN 14214 standard [8] (Table 2).

Table 1: Physical and chemical characteristics of diesel fuel and HVO according to the requirements of the HRN EN 590 standard

		COMPONENTS	
Property	**EN 590**	**HCU DF**	**HVO**
Cetane number	≥ 51.0	51.5	80.0*
Cetane index	≥ 46.0	53.4	93.4*
Density, kg/m³	820.0 – 845.0	831.0	**779.3**
Sulphur, mg/kg	<10.0	4	
PAH, % m/m	≤ 8.0	0.5	
Flash point, °C	> 55.0	66.0	80.5
Carbon residue, % m/m	≤ 0.30	0.0	<0.01
Ash content, % m/m	0.01	0.001	<0.001
Water content, ppm	≤ 200	45	14**
Total contamination, ppm	≤ 24	8	0,8
Oxidation stability, h	≥ 20		
Lubricity (wsd 1,4) at 60 °C	≤ 460	229	568
Kinematic viscosity at 40 °C	2.00 – 4.50	2.798	3.035
Distillation			
IBP, °C	-	175.0	191.0
10 % v/v recovered		204.5	249.7
50 % v/v recovered		268.5	280.3
90 % v/v recovered		334.5	292.3
% v/v recovered at 250 °C	< 65,0	41.1	10.1
% v/v recovered at 350 °C	≥ 85,0	96	98,0
95 % v/v recovered at °C	≤ 360,0	347.0	295.1
FBP, °C		354.0	299.9
Cloud point, °C	Reported	-6	-21
CFPP, °C		-7	-20

** Derived cetane number, ASTM D 6778*
*** The result was below the limit of quantification (26 mg / kg)*

Table 2: The key physical and chemical characteristics of FAME according to requirements of the HRN EN 14214 standard

	EN 14214	**FAME**
Cetane number	≥ 51.0	51.0*
Density, kg/m^3	860.0 – 900.0	882.8
Water content, ppm	≤ 500	276
Total contamination, ppm	≤ 24	7
Oxidation stability, h	≥ 8.0	8,6
Kinematic viscosity at 40 °C	3.50-5.50	4.719
CFPP, °C		-17

** Literature data*

2.3 Hydrocarbon composition of diesel fuel and HVO

The structural composition of hydrocarbons of diesel fuel and HVO was determined by comprehensive two-dimensional gas chromatography GCxGC, in-house method. The analysis confirmed mostly paraffinic composition of HVO with the main share of iso-paraffins. Results showed that hydrotreated vegetable oil is a mixture of 82.15 % m/m iso-paraffins,17.80 % m/m n-paraffins and negligible amounts of cyclo-paraffins, 0.05 % m/m. The specific chemical structure of HVO determines its behavior in blends with diesel fuel as well as diesel fuel and FAME. The tested sample of HCU diesel fuel (HCU DF) consists of 21.89 % m/m iso-paraffins, 15.70 % m/m n-paraffins, 46.76 % m/m cyclo-paraffins and 15.65 % m/m aromatics (table 3).

Table 3: Hydrocarbon composition of diesel fuel and HVO

% m/m	**HCU DF**	**HVO**
mono-aromatics	15.36	-
di-aromatics	0.29	-
three-aromatics	<0.01	-
polyaromatics	0.29	-
Total aromatics	15.65	-
n-paraffins	15.70	17.80
izo-paraffins	21.89	82.15
paraffins (n-, izo-paraffins)	37.59	99.95
cyclo-paraffins (napthenes)	46.76	0.05
Total paraffins	84.35	100.00

2.4 Properties of blends

Physical and chemical characteristics of the following blends were determined according to EN 590 standard and results are given in Table 4:

- Blend 1: 10 % v/v HVO + 90 % v/v HCU DF
- Blend 2: 18 % v/v HVO + 82 % v/v HCU DF

- Blend 3: 3 % v/v HVO+ 7 % v/v FAME + 90 % v/v HCU DF

In blends with tested diesel fuel it is possible to add maximum 18 vol. % of HVO in order to keep density within the limits given in EN 590 standard. The calculation of density was performed by commercial software. Obtained density in blends meets the expectation: density of Blend 3 > density of HCU DF > density of Blend 1 > density of Blend 2. HVO has very high cetane number (80.0). Thus, 10 % of HVO in Blend 1 significantly raised cetane number, so that it amounted 59.9. Cetane number in Blend 2 was not measured due to the mentioned limit of motor engine method at 60. Cetane number in Blend 3 was also high, 58.1. Due to high give a way of cetane number in blends with conventional diesel fuel, it is not recommended to add a high amount of HVO in standard diesel fuel, only in premium diesel fuel grade where a high cetane number is desired characteristic.

The analysis of Blends 1 and 3 shows that the values of flash point, water content, total contamination and kinematic viscosity are within the limits of the EN 590 standard. The sulphur content in blends is not determined because it is not a critical property; in diesel fuel sulphur amounts only 4 mg/kg (table 1). Regarding the distillation range, HVO in blends with diesel fuel did not influence significantly its change. IBP was slightly decreased in comparison with pure diesel fuel, while T95 and FBP were slightly changed. FAME in Blend 3 increased slightly the end of distillation. The initial values of CP and CFPP of all blends were similar and varied from -6 to -8 °C. Addition of HVO in tested blends has not influenced the improvement of low temperature properties due to the specific hydrocarbon composition, lack of n-paraffins and their specific distributions. Package of diesel fuel additives (CFI, electrical conductivity and PP) were added in Blend 1 and 3, the usual amounts. Low temperature properties in Blend 1 and 3 after the addition of additives were analyzed and one can conclude that the adding of CFI additive does not affect the CP value of diesel fuel which is in accordance with the literature data [7]. CFPP value decreased at the most in Blend 3. A short sedimentation test (SST or Aral test) that simulates winter conditions was conducted on Blends 1 and 3. The operability of blends was calculated from the CFPP value before the SST test and from the CP value after the SST. Thus, the operability value of Blend 1 (-5.1 °C) was worse than that of diesel fuel, while in Blend 3 was -11.6 °C, which is close to the limiting value of -12.1 °C, according to targeted value for CP and CFPP, and better than in pure diesel fuel. Consequently it can be concluded that CFI additive showed higher efficiency on Blend 3 than on Blend 1.

2.5 Content and distribution of n-paraffins in components and blends

Total n-paraffin content and their distribution have a high influence on low temperature properties of middle distillate. The paraffins content influences performance of the middle distillate flow improver (MDFI) additives. Sum of C_{18}-C_{25} n-paraffins is important for operability. Their low content reflects negatively on the operability. Content and distribution of n-paraffins in components and in blends was determined by gas chromatography technique, in-house method (table 4, figures 1 and 2).

Table 4: Physical and chemical characteristics of analyzed diesel fuel and its blends

		Laboratory analyses			
		HCU DF	90 % HCU DF + 10% HVO	82% HCU DF+ 18% HVO	90 % HCU DF + 7% FAME + 3% HVO
Properties	**EN 590**	**Base DF**	**Blend 1**	**Blend 2**	**Blend 3**
Cetane number	≥ 51.0	51.5	59.9		58.1
Cetane index	≥ 46.0	53.4	54.7		52.6
Density, kg/m^3	820.0-845.0	831.0	824.7	820.7	831.7
PAH, % m/m	≤ 8.0	0.5			
Flash point, °C	> 55.0	66.0	65.5		65.5
Carbon residue, % m/m	≤ 0.30	0.0			
Ash content, % m/m	0.01	0.001			
Water content, ppm	≤ 200	45	<26		43
Total contamination, ppm	≤ 24	8	1.8		2.2
Oxidation stability, h	≥ 20				
Lubricity	≤ 460		432		188
Viscosity at 40 °C, mm^2/s	2.00-4.50	2.798	2.649		2.722
Distillation					
10% v/v recovered		204.5	196.1	198.9	197.1
50 % v/v recovered		268.5	267.4	270.4	273.6
90 % v/v recovered		334.5	331.8	328.9	338.3
% v/v recovered at 250 °C	< 65.0	41.1	38.5		36.5
% v/v recovered at 350 °C	≥ 85.0	96	95.8		94.8
95 % v/v recovered at °C	≤ 360.0	347.0	347.0	345.5	350.5
IBP, °C		175.0	166.3	169.1	167.9
FBP,°C		354.0	357.9	356.5	359.9
Cloud point, °C	reported	-6	-7	-7	-6
CFPP, °C		-7	-7	-8	-6
Cloud point, °C before ARAL test*		-6	-6		-5
CFPP, °C before ARAL test*		-21	-22		-26
Cloud point, °C after ARAL test*		3	5		0
CFPP, °C after ARAL test*		-5	-4		-25
Operability, °C		-6.7	-5.1		-11.6

** Sample is additized with cold flow improvers, electrical conductivity and PP additives*

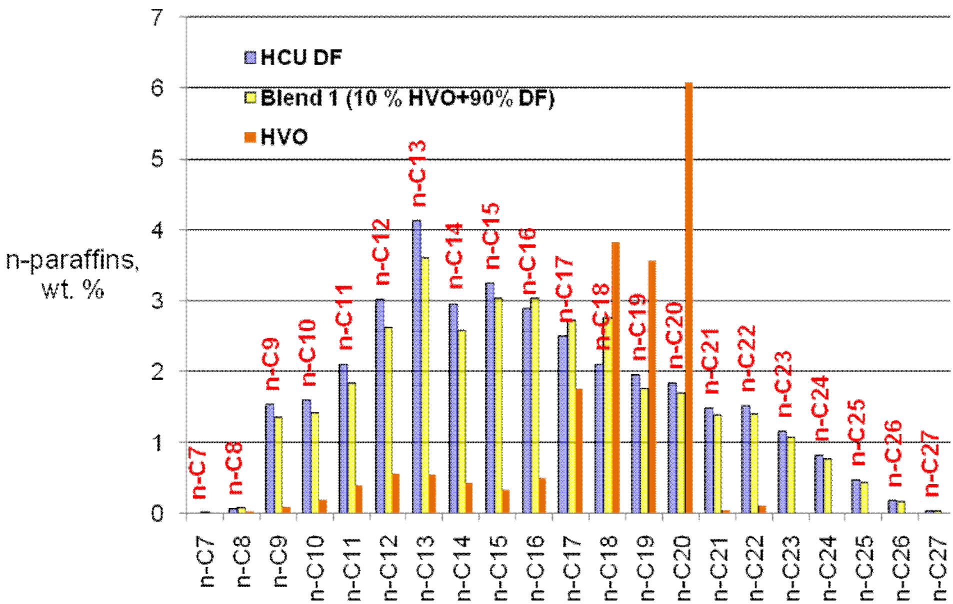

Figure 1: Comparison of n-paraffins distribution in components and Blend 1

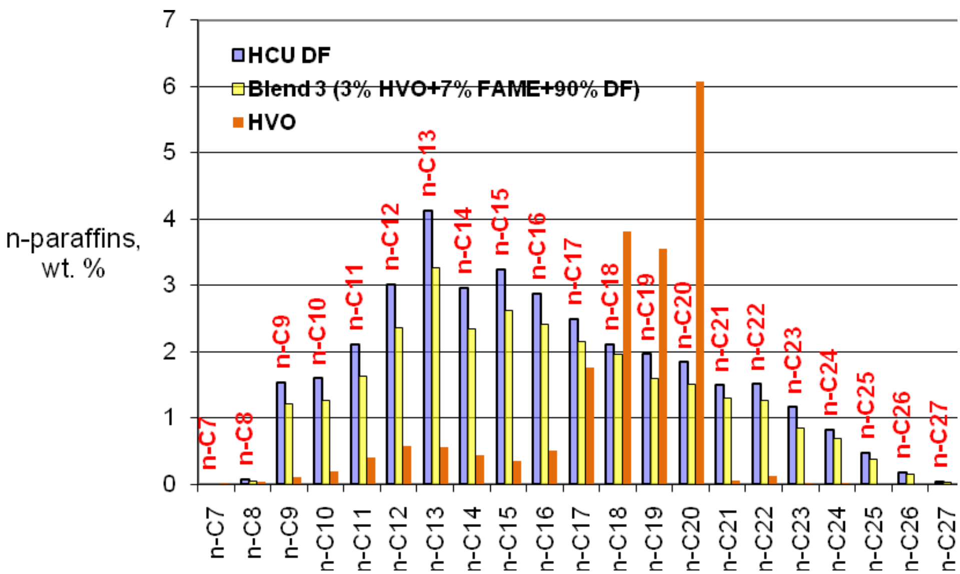

Figure 2: Comparison of n-paraffins distribution in components and Blend 3

The content of n-paraffins in HVO is about half the content in diesel fuel. The HVO's hydrocarbons are in the C_5 – C_{22} range where the main contributors are between C_{15} – C_{18}, in contrast to diesel fuel that has the wider distribution from C_8 to C_{27}.
By comparing the content and distribution of n-paraffins in Blend 1 and 3 can be concluded that higher amount of HVO in Bend 1 influenced the higher content of total n-paraffins as well as the sum of C_{18}-C_{25} n-paraffins, which resulted in the decreasing influence of MDFI additives and affected negatively the operability. Therefore, Blend 3 has better MDFI additive efficiency and better operability.

Table 5: Content and distribution of n-parrafins in components and blends

n-paraffin content	HCU DF, wt. %	HVO, wt. %	Blend 1 10 % HVO + 90 % HCU DF, wt. %	Blend 3 3% HVO + 7 % FAME + 90 % HCU DF, wt. %
n-C5	0	0.02		
n-C6	0	0.04		
n-C7	0	0.10	0.02	0
n-C8	0.07	0.20	0.08	0.06
n-C9	1.53	0.40	1.35	1.21
n-C10	1.60	0.57	1.42	1.26
n-C11	2.11	0.55	1.85	1.64
n-C12	3.02	0.44	2.62	2.37
n-C13	4.13	0.34	3.60	3.27
n-C14	2.96	0.51	2.58	2.35
n-C15	3.24	1.75	3.03	2.62
n-C16	2.88	3.82	3.03	2.41
n-C17	2.49	3.56	2.73	2.15
n-C18	2,11	6.07	2.75	1.96
n-C19	1.96	0.05	1.77	1.60
n-C20	1.84	0.12	1.70	1.51
n-C21	1.49	0.02	1.38	1.31
n-C22	1.51	0.02	1.40	1.26
n-C23	1.16	0	1.08	0.85
n-C24	0.82	0	0.77	0.69
n-C25	0.47	0	0.44	0.39
n-C26	0.18	0	0.17	0.16
n-C27	0.04	0	0.04	0.03
Total n-paraffins	35.61	18.58	33.81	29.10
Sum C_{18}-C_{25}	11.36	6.28	11.29	9.57

2.6 Comparison of properties of blends obtained by analyses and estimated by commercial software

Experimental results from analyses of Blends 1 and 3 were compared with properties obtained by commercial software which is used for the diesel fuel blending (formulation of fuel), its optimization and estimation of blends' properties. Estimated values for the density, kinematic viscosity, distillation range, flash point and low temperature properties obtained by mentioned software are in good agreement with experimental results (table 6). Estimated cetane number by software is significantly lower than measured results which implicates that it is better to determine the cetane number by other methods.

Table 6: Comparison of measured and calculated (predicted) properties of blends obtained by commercial software

		Laboratory results		Commercial software	
Properties	**EN 590**	**Blend 1**	**Blend 3**	**Blend 1**	**Blend 3**
Cetane number	≥ 51.0	59.9	58.1	54.4	52.3
Cetane index	≥ 46.0	54.7	52.6	57.5	54.5
Density, kg/m^3	820.0-845.0	824.7	831.7	825.8	833.1
Flash point, °C	> 55.0	65.5	65.5	67.5	68.9
Water content, ppm	≤ 200	13*	43	42	60
Total contamination, ppm	≤ 24	1.8	2.2	7	8
Lubricity (wsd 1,4) at 60°C	≤ 460	432	188	407	367
Kinematic viscosity at 40 °C	2.00-4.50	2.649	2.722	2.677	2.814
Distillation					
% v/v recovered at 250 °C	< 65.0	38.5	36.5	36.5	35.8
% v/v recovered at 350 °C	≥ 85.0	95.8	94.8	96.0	95.5
95 % v/v recovered at °C	≤ 360.0	347.0	350.5	341.5	346.1
IBP, °C		166.3	169.1	176.6	186.3
FBP, °C		357.9	356.5	348.6	355.6
Cloud point, °C	Report	-7	-6	-7.5	-6.0
CFPP, °C		-7	-6	-8.3	-8.1

**Result is below the detection limit (26 mg/kg)*

3. Conclusion

Based on the measured results of of pure HVO, and HVO in blends with diesel fuel, it could be concluded:

- HVO is one of the potential synthetic fuels for further use as biocomponent in diesel fuel.
- HVO is paraffinic, drop-in fuel with the following advantages: very high cetane number, low density, high energy content, sulphur and aromatic free, reasonable distillation range within diesel fuel. There are no problems during storage regarding the stability, water separation, microbiological growth and precipitation above cloud point.
- It is possible to blend maximally 18 % v/v of HVO in tested diesel fuel to meet the EN 590 standard due to low HVO density.
- Results of tested blends are in accordance with EN 590 standard.
- The characteristic low density of HVO is very interesting for INA due to the possibility to decrease high density of gas oils originated from naphthenic crudes processed in INA refineries.
- The HVO hydrocarbons composition showed a specific distribution of hydrocarbons. HVO consists of iso-paraffins component with small amount of n-paraffins, traces of cyclo-paraffins and it is completely aromatic free.
- Small amount HVO increased significantly cetane number in both types of blends. It will be possible to blend diesel fuel of premium grade with small amount of HVO.
- It could be concluded that adding of HVO does not contribute to the improvement of low temperature properties of tested blends. The addition of CFI additive showed better efficiency in blends with FAME.
- Based on results, the use of small amount of HVO (3 vol. %) in the blend with diesel fuel and FAME can be recommended because of its better application properties, such as increase of cetane number and better operability.
- The commercial software can be used successfully for HVO blending in diesel fuel and diesel fuel and FAME blending recipes to give fast information about main properties of blends. In this way it is possible to select the optimal blend recipes and to avoid the extensive laboratory work.

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