

IMPACT OF HYDROTREATED VEGETABLE OIL AND BIODIESEL ON PROPERTIES IN BLENDS WITH MINERAL DIESEL FUEL

by

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Physicochemical properties of three biofuels suitable for combustion in automotive diesel engines were compared. Two samples represented commercial hydrotreated vegetable oil (HVO) with different low temperature properties, one sample represented traditional biodiesel, fatty acid methyl esters (FAME). One HVO sample and FAME were blended into mineral diesel fuel to form two series of mixed fuels. One series represented mixed fuels containing 7, 20, 30, and 50 vol.% of HVO, the other series represented mixed fuels containing the same amount of FAME. All pure fuels as well as all prepared blends were then analyzed and evaluated. The composition and properties of HVO samples are discussed in detail as well as the influence of biocomponents on properties of mixed fuels.

Key words: *biodiesel, biofuel, diesel fuel, HVO, FAME*

Introduction

Vegetable oils are the main raw material for the production of biofuels used as renewable diesel fuels. While pure vegetable oils have only marginal significance as diesel fuels, in the form of biodiesel they are currently the dominant biofuels used in Diesel engines. Biodiesel (biodiesel, B100) consists of FAME produced by the transesterification of vegetable oils and animal fats (acylglycerols) with alcohols (mainly methanol) [1]. The technology of FAME production can be considered as matured although some efficiency improvement might be still expected (new catalyst, esp. heterogenous, further improvement of energy balance of existing production units, simplification of treatment of FAME and glycerol phases, deeper use of by-products *etc.*) [2]. There are different technologies in course for FAME production in all over the world [3]. The main features of the production technologies are shown in the tab. 1.

In comparison with mineral diesel fuel, biodiesel has a number of disadvantages due to its chemical nature. A slightly lower heating value of this fuel is only a marginal drawback. Beside aggressive impact of biodiesel on some elastomers, there is a risk of hydrolysis in the presence of water. Hydrolysis then produces free fatty acids, which cause corrosion of metallic materials. The worst disadvantage of biodiesel is perhaps its low oxidation stability caused by the presence of double bonds in unsaturated fatty acid chains bound in FAME molecules. The products of biodiesel oxidation can be substances having a lower molecular weight than biodiesel (aldehydes, alcohols, and carboxylic acids), but also high-molecular compounds.

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Table 1. Comparison of the methods for biodiesel production by transesterification [3]

Characteristics	Homogeneously catalyzed		Supercritical method	Enzyme catalyzed	Heterogeneously catalyzed
	base	acid			
Reaction conditions	60-70 °C 0.1 MPa	55-80 °C 0.1 MPa	239-385 °C >8.09 MPa	20-50 °C 0.1 MPa	60-252 °C 0.1-24 MPa
Reaction time	1-4 hours	1-8 hours	120-240 seconds	>10 hours	0.1-3 hours
FFA in feedstock	Saponified products	Methyl esters	Methyl esters	Methyl esters	Depends on catalyst, usually not sensitive
Water in feedstock	Interfere with reaction	Interfere with reaction	No influence	Depend on lipase type	Positive up to certain amount
Recovery of glycerol	Difficult	Difficult	Easy	Easy	Easy
Purification of FAME	Repeated washing	Repeated washing	None	None	Easy
Price of catalyst	Low	Low	No catalyst	High	Potentially low

These insoluble high-molecular substances form gummy deposits, which can plug the fuel system component. Oxidation products of biodiesel, which always penetrate into the crankcase of the engine, can also cause a rapid degradation of engine oil.

Another biofuel (than conventional biodiesel) can be made from vegetable oils via hydrogenation. One of the advantages of such renewable diesel production process is that it makes possible use of existing refining technology [4]. The products obtained from this process are in many ways comparable or even better than mineral diesel fuel. Hydrogenation transformation of vegetable oils into hydrocarbons, called also hydrotreating or hydrodeoxygenation, provides hydrocarbon-based biofuel called HVO, although also animal fats and used cooking oils can be used as raw material.

The first step of hydrogenation of vegetable oil involves the saturation of double bonds of hydrocarbon chains of fatty acids bounded in triglyceride molecules. Then the saturated triglycerides crack in two possible ways [5]. One reaction pathway is hydrodeoxygenation, which leads to formation of propane, six molecules of water, and three molecules of *n*-alkanes that have the same carbon number as fatty acids bounded to the original molecules of triglyceride. Since the triglycerides of vegetable oils contain mostly C₁₈ fatty acids, the product of hydrodeoxygenation is *n*-octadecane (C₁₈H₃₈). The other way of triglyceride transformation is hydrodecarboxylation, which generates propane, three molecules of CO₂, and three molecules of *n*-alkanes with a carbon number lower by one than the original fatty acids. The main hydrodecarboxylation product is therefore *n*-heptadecane (C₁₇H₃₆). An alternative reaction to hydrodecarboxylation leading to the formation of *n*-heptadecane is hydrodecarbonylation, but this reaction mechanism was not explicitly confirmed. Secondary subsequent reactions of *n*-alkanes take place at sufficient high reaction temperature, firstly isomerization of *n*-alkanes into *i*-alkanes [6-8]. Other possible subsequent reactions of *n*-alkanes include cyclization (forming cycloalkanes), aromatization (forming aromatics) and cracking (forming lighter hydrocarbons) [9, 10].

Primary HVO are hydrocarbon mixtures containing only saturated hydrocarbons. They are characteristic by high content of *n*-alkanes, particularly by *n*-heptadecane and *n*-octadecane, which usually form more than 50 % of the product. Other hydrocarbons present in primary HVO are predominantly *n*-pentadecane and *n*-hexadecane and C₁₅ to C₁₈ isalkanes [5-10]. High content of *n*-alkanes brings very high cetane number, but on the other hand, poor low

temperature properties. Cloud point and cold filter plugging point (CFPP) can reach or even exceed +20°C [11, 12]. For this reason, the commercial production of HVO is two-stage technology. The first stage is followed by the second stage that involves isomerization [13]. In isomerization step *n*-alkanes are converted to isoalkanes so that the content of *n*-heptadecane and *n*-octadecane in the final product drops dramatically. The low temperature properties are so improved, that HVO is in this respect fully comparable with mineral diesel fuel. All HVO benefits, including an extremely high cetane number or absence of aromatics, remain. The term HVO is commonly understood as the final commercial product of vegetable oil hydroprocessing regardless of the number of stages involved in the technology.

Distillation characteristics of commercial HVO belong to the boiling point range of Diesel fuel (180-360 °C). The HVO is thus an excellent non-aromatic biofuel suitable for combustion in Diesel engines. Beside this main product, smaller amount of lighter hydrocarbon fractions are formed during hydrotreating of vegetable oil (propane, naphtha, and kerosene). In contrast to biodiesel (FAME), HVO is hydrocarbon-based fuel. It means that HVO has good oxidation stability and is fully compatible with mineral diesel fuel and engine fuel systems. The properties of HVO can be even better than properties of mineral diesel fuel. Beside lower sulphur content, it is firstly high cetane number and an extremely low content of aromatic hydrocarbons. The last two mentioned parameters favorably affect not only engine performance, but also exhaust emissions (lower emissions of particulates, CO, NO_x) [14, 15]. The HVO can be used as a component of standard diesel fuel complying with EN 590 [16] or as pure paraffinic fuel complying with EN 15940 [17].

The biggest producer of HVO in Europe is the company Neste Oil (its product is sometimes labeled as NExBTL) that produces HVO in refineries located in Finland, Netherlands, and Singapore [18]. The total capacity of these units is approximately 2.5 million tons of HVO. More than one half of feedstock is waste (animal fats and waste fractions from vegetable oil refining), less part is formed by palm oil and other vegetable oils. There are also other companies producing HVO products under various brand names, for example technology Ecofining developed by Honeywell UOP and ENI (product Green Diesel) [19], or UPM (product BioVerno) [20].

The aim of this paper is to compare the properties of traditional biodiesel (FAME) and HVO and to evaluate the impact of these biocomponents on properties of diesel fuel containing them.

Experimental

Collected fuels and preparation of mixed fuels

Four basic fuels were used in this work: reference mineral diesel fuel, traditional biodiesel (FAME) and two samples of HVO with different low-temperature properties. All these four fuels were analyzed and evaluated using methods proposed bellow. The list of fuels is presented in tab. 2.

Table 2. List of fuels used

Label	Type	Origin
HVO 1	Hydrotreated Vegetable Oil	HVO made by NESTE Oil
HVO 2	Hydrotreated Vegetable Oil	HVO made by NESTE Oil
FAME	Fatty Acid Methyl Esters	Biodiesel made from rapeseed oil (PREOL)
Diesel	Mineral diesel fuel	F-class mineral diesel fuel (Unipetrol)

Biofuels HVO 1, FAME and mineral diesel fuel were used for preparation of fuel blends containing mineral diesel fuel and only one biocomponent. The HVO 1 was preferred to HVO 2

in order to use biocomponents with similar CFPP value. Petroleum diesel was blended with HVO 1 and FAME in order to prepare eight blends containing 7, 20, 30, and 50 vol.% of HVO 1 or FAME, respectively. Properties of these fuel blends were then evaluated as commercial diesel fuel according to EN 590.

Evaluation of pure fuels and fuel blends

Physicochemical properties of pure basic fuels, as well as the properties of all prepared fuel blends were determined using standard test procedures designated for diesel fuel or for petroleum products (pour point, viscosity and nitrogen content), respectively. Uncertainty of all measurements were lower than repeatability required by corresponding method. A brief overview of used test methods is shown in tab. 3.

Hydrogenated vegetable oils (HVO 1 and HVO 2) were analyzed also by gas chromatography in order to monitor the approximate content of *n*-alkanes. These measurements were carried out using a gas chromatograph Agilent 6890 equipped with a split-type injector, FID detector and a non-polar capillary column (15 m × 0.32 mm internal diameter; film thickness 0.52 μm). Nitrogen was used as the carrier gas. The following temperature program was used: 3 min at 60 °C, followed by a linear increase at 8 °C per minute to 280 °C (10 minute). Approximate content of particular *n*-alkanes was calculated from area of the respective *n*-alkane peak and the total area of the chromatogram.

Table 3. Overview of test method used

Parameter	Test method	Instrument
Density	EN 12185	Anton Paar DMA 4000
Viscosity	ASTM D7042	Anton Paar SVM 3000
Distillation test	EN ISO 3405	Normalab Analis NDI 440
Flash point	EN ISO 2719	Anton Paar PMA 5
Cetane index	EN ISO 4264	calculation
Sulphur	EN ISO 20846	Mitsubishi TS 100
Nitrogen	ASTM D4629	Mitsubishi TN 100
Polyaromatics	EN 12916	Shimadzu LC-10 set
Total aromatics	EN 12916	Shimadzu LC-10 set
Cloud point	EN 23105	Normalab Analis NTE 450
CFPP	EN 116	Anton Paar Callisto 100
Pour point	EN ISO 3016	Normalab Analis NTE 450

Results and discussion

Pure fuels

While biodiesel consists mainly of FAME derived from fatty acids having 18 and 16 carbon atoms in their molecules, HVO consists mainly of saturated hydrocarbons having 15 to 18 carbon atoms in their molecules. It reflects in relatively narrow boiling point range of both biofuels as can be seen in fig. 1. On the other hand, traditional mineral diesel fuel for moderate climate has almost linear distillation curve covering wide range of boiling points (180-360 °C). Other physicochemical properties of all evaluated pure fuels are presented in tab. 4. The HVO has very low density in comparison with FAME even mineral diesel. It is caused firstly by absence of aromatic hydrocarbons having high density. The difference between density of mineral diesel and HVO is approximately the same as the difference between density of mineral diesel and biodiesel, which is high due to its specific composition. Too low density is in fact the only parameter of HVO, which does not fulfill EN 590 for diesel fuel. All other parameters of HVO fulfill EN 590 limits with larger margin than standard mineral diesel. The HVO has many advantages in

comparison with FAME. Beside higher heating value and low water solubility, it is firstly much higher oxidation stability. Lower boiling point and lower viscosity of HVO can be also mentioned. These parameters positively influence fuel atomization in the combustion engine. Many properties of HVO are even better than properties of standard mineral diesel fuel. Beside lower sulfur content, it is firstly high cetane index and high cetane number caused by negligible content of aromatics.

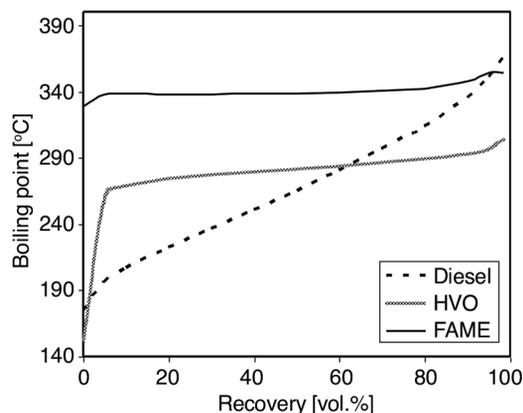


Figure 1. Distillation curves of pure fuels used for preparation of mixed fuels

Table 4. Physicochemical properties of HVO, biodiesel (FAME) and mineral diesel fuel (Diesel)

Parameter	HVO 1	HVO 2	FAME	Diesel	EN 590
Density at 15 °C [kgm ⁻³]	778.6	778.6	882.6	833.5	820-845
Viscosity at 40 °C [mm ² s ⁻¹]	2.97	2.89	4.55	2.66	2.00-4.50
Distillation					
at 250 °C recovered [vol.%]	4.8	7.3	< 0.1	39.7	max. 65
at 350 °C recovered [vol.%]	97.8 ^a	97.8	91.9	94.7	min. 85
95 Vol.% recovered at [°C]	296	295	354	351	max. 360
Flash point [°C]	75	83	170	60	min. 55
Cetane index	95	93	59	52	min. 46
Sulphur [mgkg ⁻¹]	< 2	< 2	4	8	max. 10
Nitrogen [mgkg ⁻¹]	2	2	8	34	-
Polyaromatics [% mass]	< 0.1	< 0.1	< 0.1	2.9	max. 9
Total aromatics [% mass]	0.2	0.2	< 0.1	25.0	-
Low-temperature properties					
Cloud point [°C]	-19	-34	-5	-7	-
CFPP [°C]	-23	-38	-26	-24	max. -20 ^b
Pour point [°C]	-53	<-60	-30	-30	-

^aThe final boiling point at 300-310 °C

^bF-class diesel fuel

Good low temperature properties are important in winter period and in generally in regions with cold climate. It was found, that there are HVO with various quality of low temperature properties. One type (HVO 1) had similar CFPP like standard F-class diesel fuel of (-23 °C), but relatively low cloud point (-19 °C). The other HVO (HVO 2) had CFPP similar to arctic diesel fuel (-38 °C) and very low cloud point (-34 °C). Very small difference between cloud

point and CFPP is characteristic for both HVO. All other properties of HVO 1 and HVO 2 were nearly the identical tab. 4. Different low temperature properties are probably caused by different hydrocarbon composition, particularly by different content of *n*-alkanes that have generally poor low temperature properties (high melting point). Although it is very difficult to determine individual composition of HVO by GC, *n*-alkanes can be identified and quantified for comparison purposes. A slightly different chromatographic pattern of HVO samples can be seen in fig. 2. Table 5 then presents the content of *n*-alkanes determined by GC-FID. It is obvious, that content of *n*-alkanes in HVO 1 is more than two times higher than *n*-alkane content in HVO 2. At the same time, the most abundant *n*-alkane in HVO 1 is *n*-octadecane with melting point of +29 °C. Moreover, the content of *n*-octadecane in HVO 2 is approximately three times lower than that determined in HVO 1. The difference between content of lower *n*-alkanes with lower melting point is not too significant. Low temperature properties are thus affected not only by total content of *n*-alkanes, but also by the content of particular *n*-alkanes with highest melting point. It is obvious, that the higher *n*-alkane, the higher melting point it has.

Mixed fuels

For preparation of mixed fuels, containing HVO and mineral diesel fuel, HVO 1 was used, because its CFPP value was similar to that of mineral diesel and FAME. Every mixed fuel contained only one biocomponent, FAME or HVO. The HVO may be added to mineral diesel fuel generally in any ratio. Regarding to EN 590, there is only one limiting factor-density. Low density of HVO decreases density of mixed fuel in that way, that the value of density of mixed fuel containing approximately 30 vol.% of HVO lies close to the lower limit 820 kg/m³, fig. 3. The precise value of limit HVO content depends firstly on density of mineral diesel fuel used for blending. The dependence of mixed fuel density on the content of HVO is similar like the dependence of density on FAME content. The only difference is that HVO decreases, while FAME increases density of mixed fuel. Density of mixed fuel containing approximately 30 vol.% of FAME thus attacks the higher limit 845 kg/m³. This is the reason, why EN 16709 [21] for high FAME diesel fuel (B20 and B30) sets the higher limit for density to 860 kg/m³ instead of 845 kg/m³.

Except density, all parameters of mixed fuels containing any ratio of HVO fulfilled EN 590 limits for diesel fuel or it can be expected. The only problematic parameter could be lubricity that could be improved with additives. Mixed fuels containing HVO have even better proper-

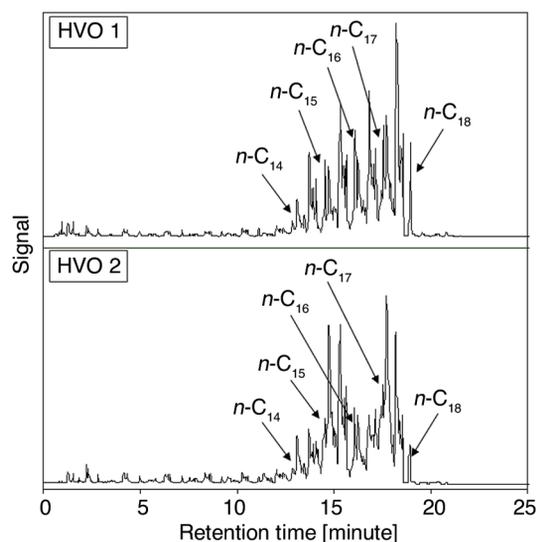


Figure 2. Chromatograms of hydrogenated vegetable oils HVO 1 and HVO 2

Table 5. Content of *n*-alkanes in HVO 1 and HVO 2 [% mass]

<i>n</i> -alkane	HVO 1	HVO 2
< <i>n</i> -C ₁₅	1.00	1.15
<i>n</i> -C ₁₅	1.38	0.54
<i>n</i> -C ₁₆	1.62	0.88
<i>n</i> -C ₁₇	1.51	0.61
<i>n</i> -C ₁₈	2.36	0.78
<i>n</i> -C ₁₉	0.02	0.01
<i>n</i> -C ₂₀	0.03	0.01
> <i>n</i> -C ₂₀	0.01	-
Total	7.92	3.69

ties than pure mineral diesel fuel. The presence of HVO in fuel has no influence on viscosity, fig. 4. The influence of HVO on flash point is relatively small and favorable, fig. 5. Addition of HVO/FAME has also favorable effect on content of aromatics. The content of polycyclic aromatic hydrocarbons and total aromatics linearly decreases with increasing content of HVO/FAME in mixed fuels. The content of these hydrocarbons can be easily calculated from composition of pure fuels and the ratio of HVO/FAME and mineral diesel in mixed fuel. In contrast to FAME, presence of HVO strongly affects cetane index as can be seen in fig. 6. Similar increase can be expected also in the case of cetane number.

Figure 7 shows the dependence of cloud point on the content of biocomponent. It was expected

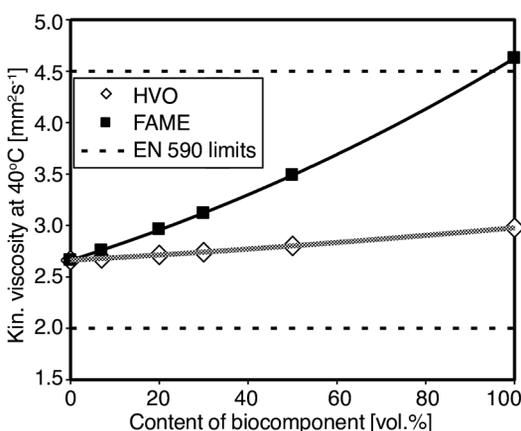


Figure 4. Viscosity of mixed fuels in dependence on the content of the biocomponent

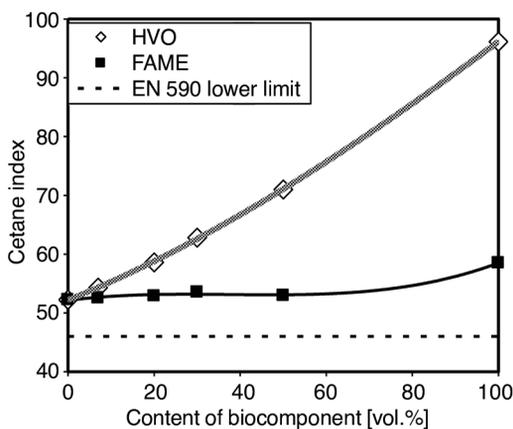


Figure 6. Cetane index of mixed fuels in dependence on the content of the biocomponent

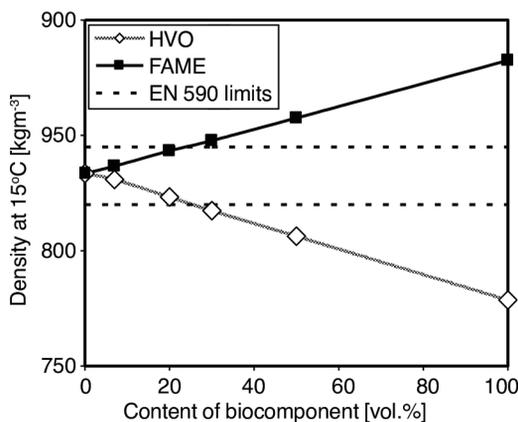


Figure 3. Density of mixed fuels in dependence on the content of the biocomponent

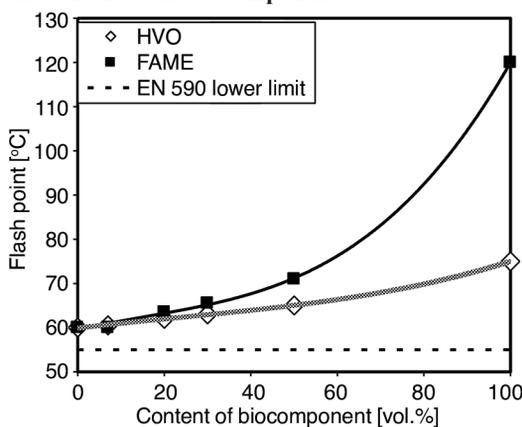


Figure 5. Flash point of mixed fuels in dependence on the content of the biocomponent

that cloud point of mixed fuels containing FAME would not be significantly affected by FAME content, because the difference between cloud point of pure mineral diesel and pure FAME is only 2 °C. The difference between cloud point of mineral diesel and HVO (HVO 1) is 12 °C and thus the dependence of cloud point on HVO content is more significant. Nevertheless, practically no change in cloud point was observed between pure mineral diesel and fuels containing 10 and 20 vol.% of HVO. The CFPP of mineral diesel and HVO was nearly the same and also all blends containing these two components did not show any unexpected behavior,

fig. 8. On the other hand, the dependence of CFPP on FAME content showed an anomaly with clear minimum of CFPP observed at 20 vol.% of FAME.

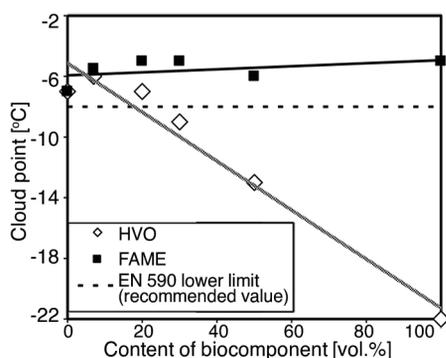


Figure 7. Cloud point of mixed fuels in dependence on the content of the biocomponent

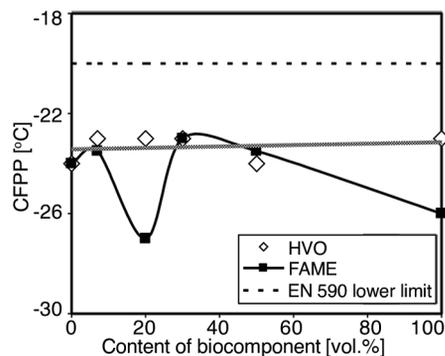


Figure 8. The CFPP of mixed fuels in dependence on the content of the biocomponent

Conclusions

Hydrotreated vegetable oil is an excellent biofuel suitable for combustion in modern automotive Diesel engines. In contrast to traditional biodiesel (FAME), saturated hydrocarbon nature of HVO brings to it better oxidation stability and high heating value. It was found that HVO can be blended as a premium biocomponent to mineral diesel fuel in much higher concentration than FAME. While FAME content in diesel fuel (EN 590) is limited by 7 vol.%, content of HVO is not directly limited. The HVO can be blended into mineral diesel fuel up to concentration of 30 vol. % in order to meet the lower limit for density required by EN 590. It was found, that relatively low density is the only one parameter that limits content of HVO in diesel fuel. Most of other HVO parameters increases diesel fuel quality with increasing HVO content definitely.

Cetane index of diesel fuel containing 30 vol.% of HVO was higher by 11 units than cetane index of mineral diesel fuel. Similar increase in cetane number can be expected. Other parameters of diesel fuel were also favorable influenced by the presence of HVO (*e.g.* flash point, sulphur content, aromatics content, monitored distillation points or cloud point). Low-temperature properties of one tested HVO were so excellent, that it could be used as a biocomponent for arctic diesel fuel. The HVO thus can be effectively used as a diesel fuel blending component in order to increase share of energy from renewable sources and reduce even more GHG without any harmful impact to modern diesel engines.

The FAME and HVO production and their use have not only specific pros and cons mutually, however, also in comparison with mineral diesel fuel. Therefore, important technological, technical, economic, product quality, environmental, social, toxicological and human health risk issues related to their production and use must be considered. Both products (FAME and HVO) have certain cost production disadvantage compared to fossil diesel fuel. The possible solution could be orientation towards low-cost and non-edible feedstock (including waste and used fats and oils), advanced technologies with reduced overall production costs, maximisation of use of by-products, improvement of product yield and economy of scale.

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