

Annex 45



A Report from the IEA Advanced Motor Fuels Implementing Agreement

Synthesis, Characterization, and Use of Hydro-Treated Oils and Fats for Engine Operation

Benjamin Stengel
University of Rostock, Germany

Johan Hardang Vium
Danish Technological Institute, Denmark



Annex 45



A Report from the IEA Advanced Motor Fuels Implementing Agreement

Synthesis, Characterization, and Use of Hydro-Treated Oils and Fats for Engine Operation

Benjamin Stengel
University of Rostock, Germany

Johan Hardang Vium
Danish Technological Institute, Denmark



March 2015

Acknowledgment

This project is made in cooperation with the International Energy Agency – Advanced Motor Fuels Agreement. The report is a contribution to Annex XXXXV, “Synthesis, Characterization and Use Hydrotreated Oils and Fats for Engine Operation”.

The project has been commissioned by the University of Rostock (Germany) as the operating agent. The University of Rostock (UR, Germany), the Danish Technological Institute (DTI, Denmark), Novozymes (Denmark), Scania DK (Denmark), CanmetENERGY (Canada), Blue Sun (USA), Neste Oil (Finland) participated on task sharing basis. The University of Rostock was responsible for the execution of engine tests, DTI was responsible for fuel obtainment and analysis of process costs and sustainability, CanmetENERGY participated through performing fuel analyses. Blue Sun and Neste Oil have been commissioned by DTI respectively UR to provide fuel for fuel analyses and engine tests.

The authors would like to acknowledge the IEA-AMF Executive Committee for supporting this Annex and the Danish Energy Agency program (EUDP) and the German Agency for Renewable Resources (FNR) for funding this research project.



Table of Contents

Acknowledgment	i
Acronyms and Abbreviations	iii
1. Introduction.....	1
2. Background	2
3. Obtainment of fuels	3
4. E-FAME and HVO process cost	4
5. Sustainability (energy cost, catalyst)	6
5.1. Production method	6
5.2. Physical properties	8
5.3. Environmental aspect	9
5.4. Sales of byproducts	11
6. Fuels analysis.....	12
7. Engine tests.....	13
7.1. Experimental design	13
7.2. Standard ECU operation	16
7.2.1. Impacts on combustion process.....	16
7.2.2. Impacts on engine control parameters.....	18
7.2.3. Impacts on emissions	22
7.3. Open ECU operation	26
7.3.1. Impacts on fuel efficiency and emissions.....	28
8. Conclusion.....	31
9. List of Figures	32
10. References	34
Appendix.....	35

Acronyms and Abbreviations

AMF	Advanced Motor Fuels
BMEP	brake mean effective pressure
CFPP	cold filter plugging point
CO	carbon monoxide
CO ₂	carbon dioxide
DOC	diesel oxidation catalyst
DPF	diesel particulate filter
ECU	engine control unit
E-FAME	enzymatic fatty acid methyl ester
EGR	exhaust gas recirculation
FAME	fatty acid methyl ester
FFA	free fatty acids
FI ^{2RE}	flexible injection and ignition for rapid engineering
FSN	filter smoke number
HRR	heat release rate
HVO	hydrotreated vegetable oil
IEA	International Energy Agency
M	engine torque
n	engine speed
NO _x	nitrogen oxides
SCR	selective catalytic reduction
THC	total hydro carbon
VTG	variable turbine geometry

1. Introduction

Due to limited crude oil stocks and a rising environmental awareness in the society the car and fuel industries are forced to develop alternative fuels. Those especially need to be profitable and producible in a sufficient amount. Moreover, vehicles using alternative fuel should have a low carbon dioxide emission and a range comparable to conventional fuels. On the way towards a CO₂-free production of fuels the use of biomass offers a possibility to produce CO₂-neutral regenerative fuels. The European Commission introduced a schedule for promoting biofuels. According to this schedule, 10% of the conventional fuels in Europe shall be replaced by biofuels by the year 2020. Currently transesterificated vegetable oils (fatty acid methyl ester, FAME) are mostly used. For production of FAME high amounts of chemicals are necessary. By the use of enzymes acting as bio catalysts the production costs could be reduced since less chemicals are needed. Moreover, a production stage could be skipped because free fatty acids do not need to be removed. FAME that is produced with the use of enzymes is called enzymatic FAME (E-FAME).

In general FAME tends to dilute the engine lubrication oil, especially when particulate filters are frequently regenerated. This may be critical when operating permanently with biodiesel. Consequently the admixture of biodiesel to conventional diesel is limited so far. An alternative may be the use of hydrotreated vegetable oils. To achieve fuel properties comparable to conventional diesel the vegetable oil not only needs to be hydrotreated but also shortened relating to their C-chain length. The final product is called hydrotreated vegetable oil (HVO). HVO is characterized by a high cetane number and a boiling behavior similar to diesel. In contrast to FAME no incompatibilities against varnishes and sealing materials are apparent and HVO does not tend to dilute lubrication oils. Furthermore, HVO shows a better emission behavior especially in relation to soot compared to standard diesel, which is caused by the absence of aromatic compounds and a better mixture formation. HVO is miscible with conventional diesel and can also be used as a straight diesel substitute. Production of HVO is mainly based on palm oil and waste like animal fats as feedstocks [1].

2. Background

The conventional use of fossil fuels for passenger car and ship engines will persist for the upcoming decades, even though the costs for oil will increase. However, as a result of emissions from internal combustion engines and limited oil stocks, it is necessary to conduct research on alternative fuels.

The introduction of diesel emission standards during the last decades led to the development and implementation of exhaust after treatment systems like DOC, DPF or SCR catalysts, Figure 1. Former studies [2] showed an emission reducing potential of biofuels. Thus biofuels may help meeting upcoming emission standards.

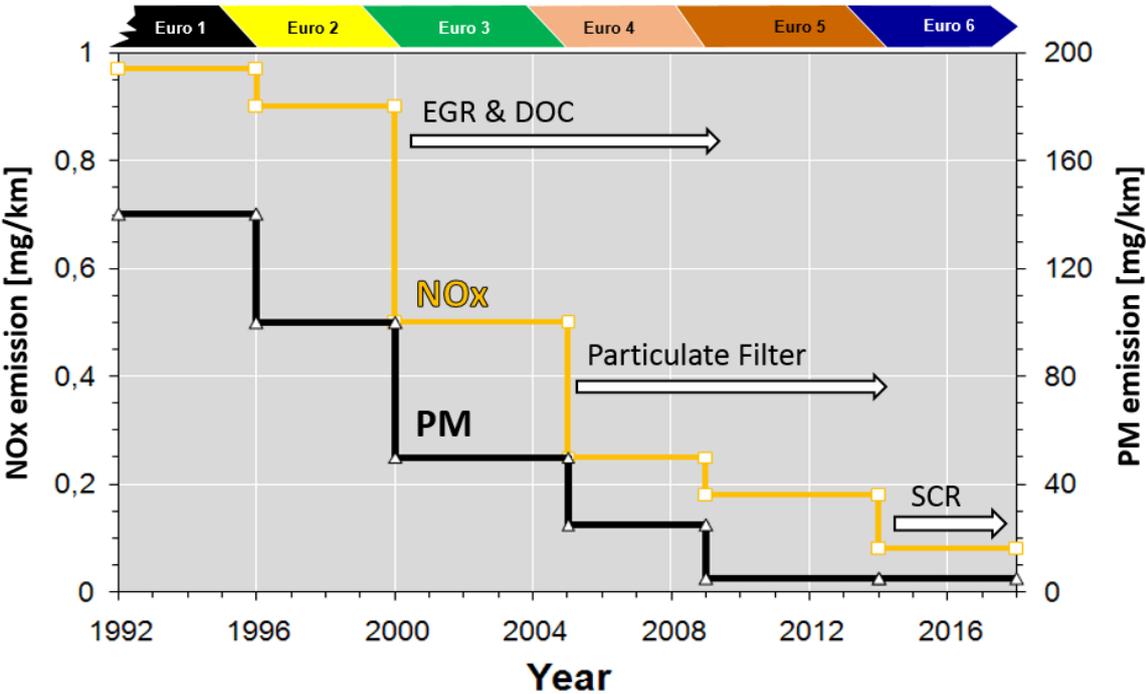


Figure 1: Evolution of European emission standards for passenger car diesel engines, [3]

Currently vegetable oils, in connection with transesterification (fatty acid methyl esters, FAME), are used mainly for blending with fossil diesel, Figure 2. However, increased FAME blends can lead to the dilution of lubrication oil, thereby resulting in shorter intervals between oil changes. Furthermore fouling can occur, which may lead to damage of high-pressure injection pumps.

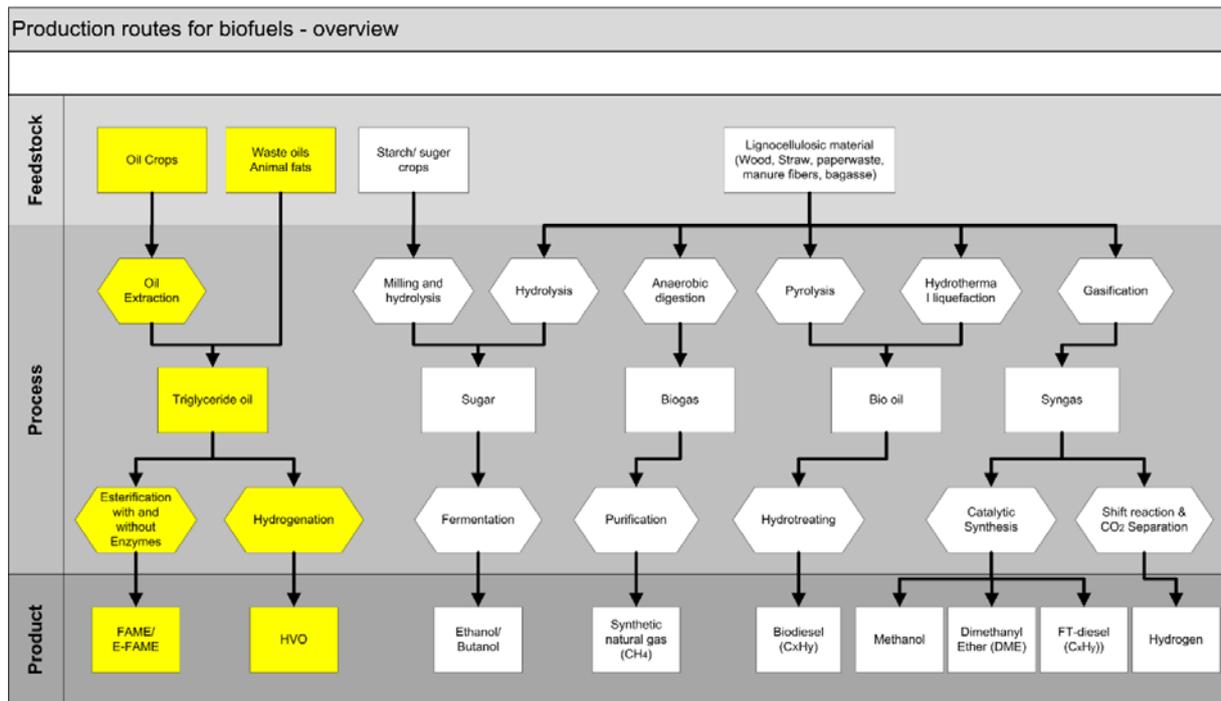


Figure 2: Schematic of different routes to biofuels. The specific fuel and fuel routes tested and analyzed in this project is highlighted in yellow

One promising alternative is the use of hydrotreated vegetable oil (HVO) as a diesel substitute or blend component. The HVO shows very similar characteristics compared to conventional diesel and results in reduced exhaust emissions [2]. Further research is needed in order to reduce the high production costs of HVO. Simultaneously, the production method for FAME must be improved to decrease costs.

This study investigates the use of E-FAME and HVO as straight diesel substitutes in modern diesel engines and their process costs and sustainability.

3. Obtainment of fuels

800 Liters of Enzymatic-FAME was obtained from Blue Sun at their factory in St. Joseph Missouri, USA. The Danish company Novozymes that produces the enzymes for the production process helped obtaining and transporting the fuel to the test laboratory at University of Rostock. The feedstock used for the fuel consists of approximately 80% corn oil from bioethanol plants and 20% from used cooking oil. The enzyme used is a lipase from Novozymes Callera™ Trans. As a pretreatment, before

the actual biodiesel transesterification process, Enzymes are used for reducing free fatty acids. The pretreated oil is transesterified, and the FAME phase is separated from the glycerin phase, distilled, and finally washed with NaOH to achieve the final product.

600 Liters of NExBTL was obtained from Neste Oil Corporation in Finland. NExBTL is the trade name for HVO produced by Neste Oil. NExBTL is based on several different feedstocks. According to [1] 50% of the raw material is derived from palm oil and 50% from waste such as animal fats.

4. E-FAME and HVO process cost

To establish cost of the different processes it is important to acknowledge that different feedstock has different properties, Figure 3. Some oils contain large amounts of free fatty acids, which is prohibiting for especially the standard biodiesel process. For example, animal fat and used butter fat has large amounts of FFA (5-30 %), but is also a cheaper feedstock.

In addition, the amount of saturated and unsaturated fat varies; this creates different fuels in the end when using the FAME biodiesel transesterification process.

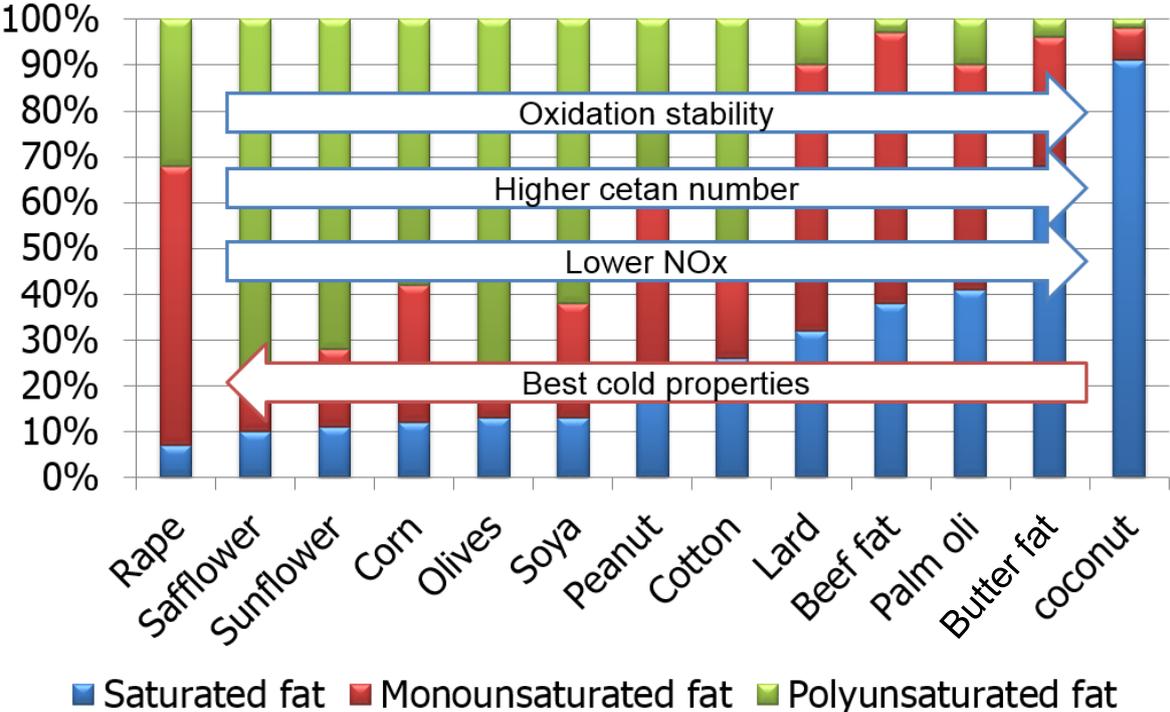


Figure 3: Overall properties of FAME biodiesel depending on source of bio oil

The price of the different feedstock also varies, and gives different possibilities of producing. Some require substantial preparation before the actual biodiesel production process. This preparation can be done either chemically or by using enzymes. The E-FAME tested in this project is produced partly using enzymes from Novozymes. The hydrotreated vegetable oil process is not as dependent on the feedstock. Here it is possible to remove unwanted double bonds by adding hydrogen.

The biodiesel prices in Europe are also influenced by the origin of the biodiesel. In Europe, there is a set mixture of 5.75% by energy content biofuel in diesel sold at gas stations. Here biodiesel produced from 2nd generation biofeedstock, such as waste butter fat and dead animal, counts twice as much as biodiesel produced from regular oil crops such as rape, corn or palm. This means that adding 2.875% by energy of 2nd generation biodiesel to standard diesel meets the demand of 5.75% 1st generation biofuel. Distillation and washing with for example NaOH before selling the fuel contributes to the uniformity and quality of the fuel, but also cost of the fuel.

Due to the high content of FFA in animal fat and plant oils the use of enzymes in the FAME process for this production is the most likely. This is due to the inhibiting process and the production of soap in the traditional FAME process when the FFA content is high. The current production of enzymatic based FAME is based on this feedstock. Another challenge for comparing the different fuel production prices is that production plants for HVO biodiesel typically are 10-50 times larger than production facilities for FAME. For E-FAME, there are only few plants operating and not sufficient data has been available for a thorough analysis, so it is based on different estimations.

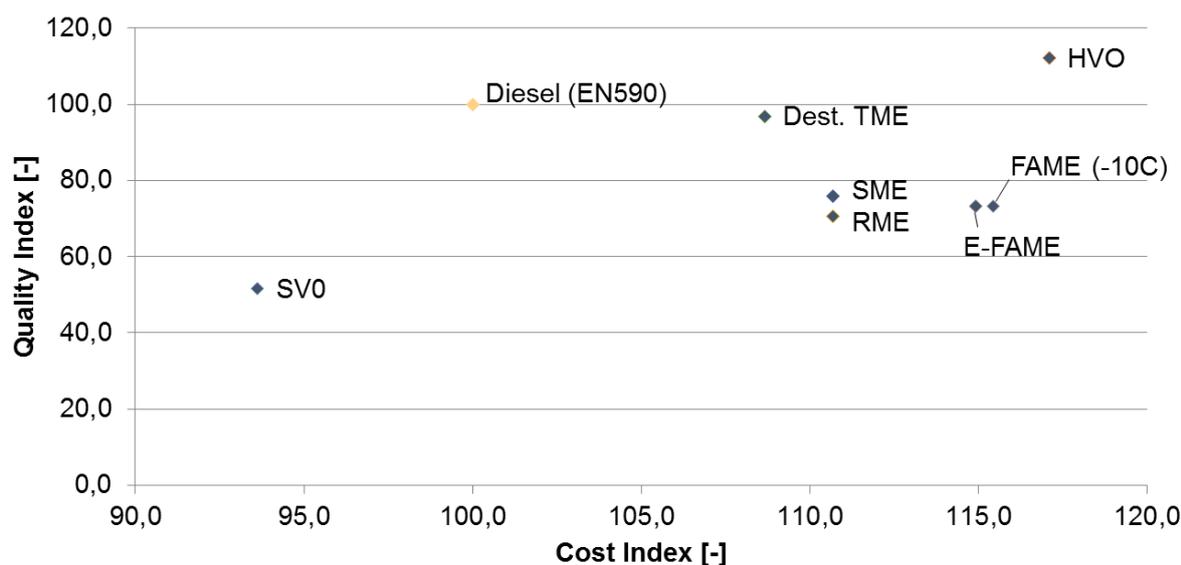


Figure 4: The quality index is based on cetane number, storage stability and CFPP (Cold Filter Plugging Point) of the fuels, the cost index is based on different sources and estimations

5. Sustainability (energy cost, catalyst)

5.1. Production method

For better understanding of the background of the alternative diesels' properties, the production principles are roughly described. A schematic comparison of the production methods can be found in Table 1.

FAME biodiesels are produced using a process called transesterification, where alcohol is added to animal fat or vegetable oil using a catalyst. The process reforms the oil or fat molecule into ester molecules and glycerin. The catalyst, glycerin and excess alcohol are separated from the FAME after the process has ended. There are several different catalysts for producing FAME, the most common used are alkaline but recently enzymes are also used. If the catalyst used is an alkaline, it is neutralized using acid forming salt and water. The salt is then separated from the rest and is either treated as waste or sold as fertilizer, depending on the alkaline used.

Enzymes are a more expensive catalyst, but it can be reused. The very expensive immobilized enzyme can be reused up to 100 times while the cheaper liquid enzyme can be reused on average three times. There is no need for neutralization or subsequent salt removal as in the alkaline process.

Depending on the purity, the byproduct, glycerin, is either sold for chemical uses or used as animal feed. Using enzymes as catalyst typically gives a higher purity of glycerin.

If alkaline treatment is used for transesterification of animal fat or other oils containing a high amount of free fatty acids, the oil has to be pretreated before the alkaline is added. No pretreatment is needed when enzymes are used as catalyst.

HVO (Hydrotreated Vegetable Oil) biodiesels are produced using hydrogen treating. During the process, hydrogen is added to animal fat or vegetable oil using a metal catalyst. The catalyst can be reused almost indefinitely. This process produces paraffin and the byproducts water, propane and CO₂. Paraffin is the preferred diesel fuel component for combustion engines due to its high cetane number. HVO is also preferred due to lower sulfur or instability issues. Also see the chapter on tests of the different biofuels in this project.

After the paraffin has been separated, the product can either be sold or after treated by isomerization, to improve cold properties.

Table 1: Comparison of production methods

	Traditional FAME (alkaline)	Enzymatic FAME (liquid)	HVO
Feedstock	Vegetable oil/animal fat/Waste cooking oil	Vegetable oil/animal fat/Waste cooking oil	Vegetable oil/animal fat/Waste cooking oil
Secondary feedstock	Methanol	Methanol/ethanol	Hydrogen
Use of secondary feedstock kg/kg	0,142 kg/kg	0,1 kg/kg	0,015 kg/kg
Catalyst	Sodium hydroxide: 3,11g/kg Phosphor acid: 2,44g/kg	Callera Trans L 20g/kg 3 cycles	NiMo
Process	Esterification /Transesterification	Esterification/Transesterification	Hydrogen treating/ Isomerization

5.2. Physical properties

The quality of the diesel product, in terms of performance and environmental impact, greatly depends on the physical properties. Alkaline FAME and enzymatic FAME have very similar properties, while HVO is different. In Table 2 the properties are compared. There are typically also big differences depending on which feedstock is used for the production of the FAME biodiesel.

The density of both HVO and FAME is outside of the allowed range of standard diesel (EN590), FAME higher and HVO lower. The density of the fuel is a main characteristic used for defining if it is diesel, gasoline etc.

The heating value of FAME is somewhat lower than standard diesel, meaning more FAME is needed in order to get the same amount of energy. HVO however, has a bit higher heating value.

The cetane number of the fuel is perhaps the most important property for a diesel engine. It defines the ignition delay of the fuel. The European standard for diesel states that this must be above 51. Typically FAME has a cetane number of 61 while HVO has a cetane number of 88.

HVO are in several ways superior to FAME, making it a higher value product. The diesel standard EN 590 allows for a blend of 7% of FAME in the diesel. HVO should be able to blend in standard diesel in any ratio [4] as long as the blend density stays within the applicable limits.

Table 2: Comparison of properties [5]

	[Unit]	Standard diesel	Alkaline FAME (rapeseed)	Enzymatic FAME (rapeseed)	HVO
Density	Kg/m ³	820-845	876	880	780
Heating value LHV	MJ/kg	43.4	37.4	37.2	44.1
Heating value LHV	MJ/l	36	32.8	32.7	34.4
Cetane number		>51	61	>51	88
Viscosity	mm ² /s	2-4.5	4.54	2-4.5	3.0
Poly aromatics	% (m/m)	<8	<1	<11	<0.1
Sulfur	mg/kg	<10	8	<10	<5.0
Flash point	°C	>55	>55	>55	>61
Distillation 95% (v/v)	°C	<360		<360	<320
Cold Filter Plugging point	°C	(-22)-(-44) ¹	-15	-15	-32

5.3. Environmental aspect

During production of both alternative diesel fuels and fossil diesel fuel, energy is used and greenhouse gases are emitted. In the report by Edwards et al. (2014) [5], the energy use and greenhouse gases, during the whole production, are listed for standard diesel, HVO and FAME. The energy use and greenhouse gas emissions used in this chapter are listed in Table 3. Figure 5 shows an overview of the energy use and greenhouse gas emissions from each production method.

Since this survey deals with refining processes, the GHG emitted from final fuel combustion is not included. The energy associated with the cultivation and harvesting of oil crops, and fossil oil production and transport to refineries are not included.

The reformation of crude oil into standard diesel is relatively simple compared to FAME and HVO production. The refining of crude oil uses less energy, but emits more

¹ Dependent on region

greenhouse gasses because mainly fossil sources of energy are used for the reformation process.

The publication by Edwards et al. (2014) [5] shows that hydrogenation of vegetable oil or animal fat into HVO requires less energy than the alkaline transesterification into FAME. This is mainly due to a simpler process with fewer steps. The publication also states that the greenhouse gases (measured in equivalence to CO₂) for both HVO and FAME is in the same range.

A source for a large difference in greenhouse gas emissions during production is the source of methanol and hydrogen. Most of the methanol and hydrogen are currently extracted from crude oil, but there is a great potential for using renewable sources, reducing the greenhouse gas emissions further.

Since the use of enzymes as catalyst for FAME production is a relatively new technology, no actual data from an industrial scale plant is provided. By comparing the alkaline transesterification to the enzymatic transesterification process, however, an estimation of the greenhouse gasses and energy use can be made.

The greenhouse gas emission from the two enzymatic processes are presumed to be in the same range, because the processes are very similar. The enzymatic FAME, made from rapeseed, is assumed to emit slightly less greenhouse gasses than using alkaline, because it is a slightly simpler process. Animal FAME processed by the enzymatic route is presumed to have lower greenhouse gas emissions than if processed by alkaline. This is because the glycerin from enzymatic FAME made from animal fat is of higher value and have higher yield. In the model, glycerin is counted as a byproduct and thereby account for a part of the greenhouse gasses. When the yield and value of the glycerin is higher, the percentage of the greenhouse gasses it accounts for rises.

The energy use during production of the enzymatic FAMEs are expected to be lower than for their alkaline counterparts. This is because that there is no need for pretreatment due to free fatty acids, and the after treatment are less energy consuming.

Table 3 The energy use during production of 1 MJ fuel, values are found in Edwards et al. [5]

		Standard diesel	FAME Rapeseed Alkaline	FAME Rapeseed Enzyme	HVO Rapeseed	FAME Animal fat Alkaline	FAME Animal fat Enzyme	HVO Animal fat
Energy use during production	MJ/MJ _{fuel}	0,1	0,14	0,135	0,14	0,135	0,145	0,11
Greenhouse gas emissions during production	GHG g CO ₂ /MJ _{fuel}	8,6	3,9	3,8	6,8	7,04	4,5	5,3

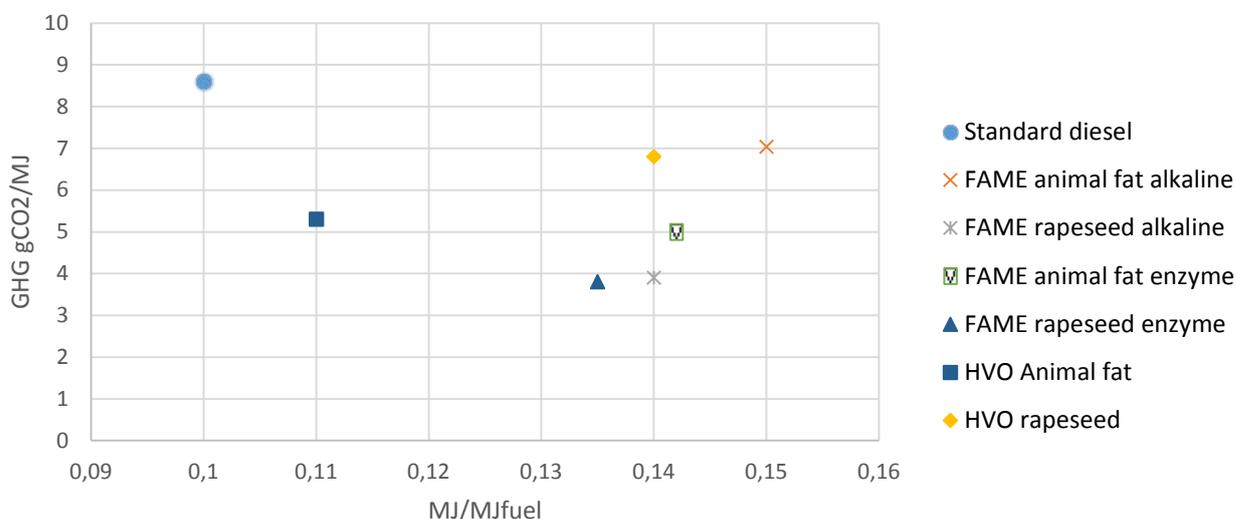


Figure 5: Overview of energy use and GHG emissions during the production of 1MJ fuel (LHV)

5.4. Sales of byproducts

When producing alternative diesels, byproducts are generated. To bring down the production cost of the fuels these byproducts can be sold or used in the plant.

When producing FAME a byproduct of glycerin is generated. This glycerin can be sold either for chemical use, or as animal fodder. If the glycerin is sold for chemical use, it is worth up to 10% of the FAMEs value. If enzymes are used for the FAME production, the glycerin are typically of higher value than if it is produced by using alkaline as catalyst.

Enzymes for FAME production are more expensive than alkaline, however where the alkaline is only used once, enzymes can be reused several times.

The byproducts from HVO production are water, CO₂ and propane. The propane is usually used for heating the plant, reducing the amount of energy needed for production of HVO. The production cost of HVO is typically slightly higher than that of the FAME.

6. Fuels analysis

Fuels used for engine tests were European standard diesel (EN 590), HVO and E-FAME. Fuel analysis was organized by CanmetENERGY (Canada) and performed by Alberta Innovates – Technology Futures (AITF). The full report of analysis can be found in Appendix A. Fuel analyses were performed according to EN 590 specification for the reference diesel and HVO respectively according to EN 14214 for E-FAME. In accordance with fuel properties from literature survey neither the density of HVO nor of E-FAME meet the specification of standard European diesel EN 590. However earlier studies showed that both HVO and FAME can be used as standard diesel blends while meeting EN 590 limits [2]. Moreover, cetane numbers were determined. HVO's cetane number is significantly higher compared to standard diesel suggesting a decreased ignition delay. E-FAME's cetane number is slightly lower than that of diesel indicating an increased ignition delay. Further analysis performed at the Laboratory of Operating Supplies (University of Rostock, Germany) shows decreased heating values of E-FAME and FAME while HVO's heating value is slightly higher compared to standard diesel (Table 4). The lower the fuel's heating value the higher amount of fuel needed for the same energy output.

Table 4: Lower heating values of EN 590 diesel, HVO, E-FAME and FAME

Parameter	Unit	Standard diesel (EN590)	HVO	E-FAME	FAME
Cetane number	[-]	54	79	51	64
Heating value	[kJ/kg]	42517	43846	37309	37410

7. Engine tests

7.1. Experimental design

In order to investigate the influences of fuel properties on combustion and emissions steady state experiments were performed using a modern EURO-VI passenger car diesel engine. The test engine was equipped with sensors for monitoring temperatures, pressures and mass flows. It was first operated with its standard ECU using a four quadrant brake (AVL INDY P-22) at selected operation points (Figure 6).

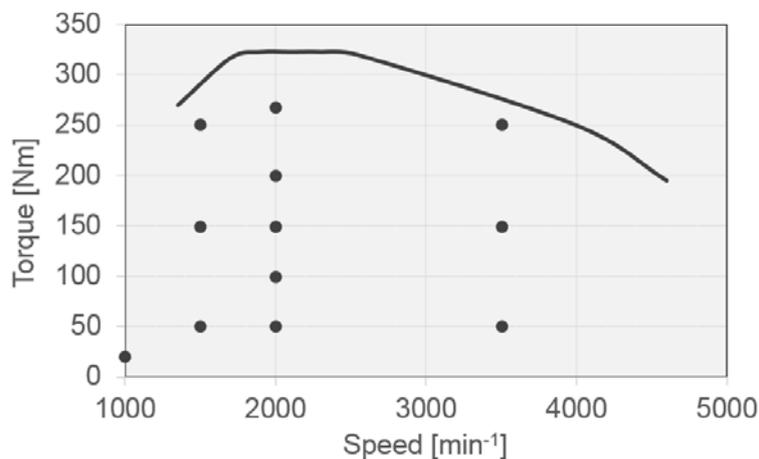


Figure 6: Selected operation points for fuel testing

An AVL SESAM-IV system was used to measure gaseous emissions. For determining soot emissions an AVL Smokemeter was used. The efficiency of exhaust after treatment components could be measured by installing exhaust sampling points both before and after each component. An indication system (Kistler pressure sensor 6043 and Kistler KiBox) allowed analysis of in-cylinder pressure traces. Furthermore heat release rates could be calculated based on in-cylinder pressure traces using a

thermodynamical single-zone model. A clip on ampere meter was attached to record the current feed to the engine's injectors. Moreover ECU data could be readout and recorded using an ECU-PC interface (ETAS ES 590 and ETAS INCA).

Additionally to the engine tests with the standard ECU, experiments with an open ECU were performed in the second step. For this purpose the application tool IAV FI^{2RE} was connected to the engine. This tool offers multiple current feeds of injectors as well as control of the actuators for throttle, EGR-valve, VTG and the control of the fuel rail pressure. Thus fuel influences could be selectively analyzed while varying engine control parameters.

The test engine used for the experiments was a 2.0 liter turbo diesel engine from Volkswagen. This engine represents the current state of the art by the use of its comprehensive exhaust after treatment (DOC, DPF, SCR – Figure 7) and its modern combustion processes. Furthermore a second generation high pressure CR-system is equipped to the engine allowing a flexible control of multiple injections per combustion cycle which makes the engine very suitable for investigating the potential of biofuels. Table 5 shows the main technical parameters of the test engine.

Table 5: Specifications of the test engine

Test engine	VW Passat CBAC
Displacement	2.0 liter / 1,968 cm ³
Engine type	4 – cylinder, 16 valves
Rated output	103 kW at 4,200 min ⁻¹
Max. torque	320 Nm at 1,750 – 2,500 min ⁻¹
Stroke	95.5 mm
Bore	81.0 mm
Injection	Common-Rail-Injection (second generation)
Exhaust after treatment	DOC – DPF – SCR
EGR	Cooled high-pressure EGR
Turbocharger	Turbocharger with VTG

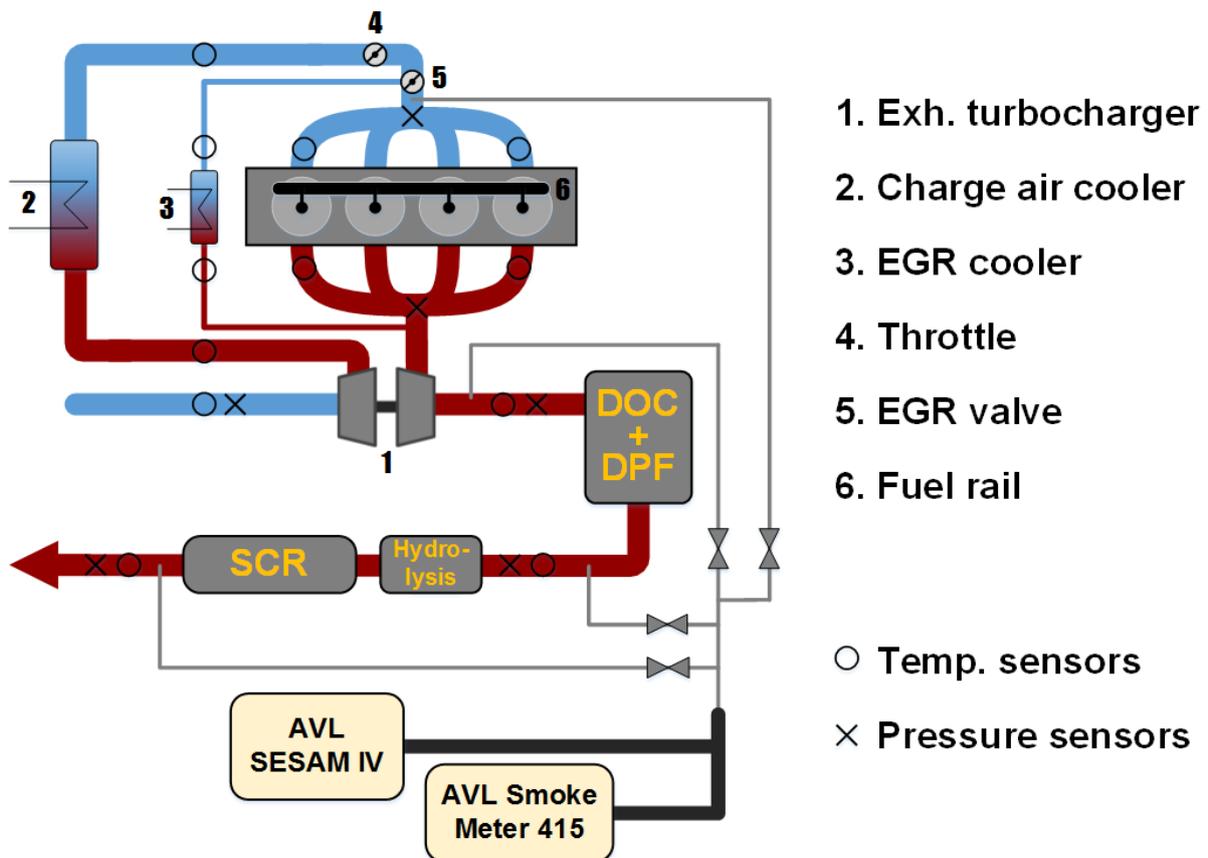


Figure 7: Scheme of the test engine



Figure 8: Test engine with exhaust after treatment components and test bench brake

7.2. Standard ECU operation

The test engine was first operated with its standard ECU at selected operation points (Figure 6) and analyzed according to impacts on combustion process, engine behavior and emissions.

7.2.1. Impacts on combustion process

In order to analyze the impacts on the combustion processes in-cylinder pressure traces were measured and processed to calculate the heat release rates, Figure 9. The HRRs show two distinct pre-combustions resulting from two pre-injections. This technique is used for pre-conditioning of the combustion chamber in order to decrease pressure gradient of the main combustion. Thus, noise emissions and NO_x emissions could be reduced. The HRR of HVO is characterized by an earlier ignition of the first pre-combustion compared to the other fuels reasoned by its increased cetane number. The main combustion of HVO starts slightly delayed and with higher heat release compared to EN 590 diesel.

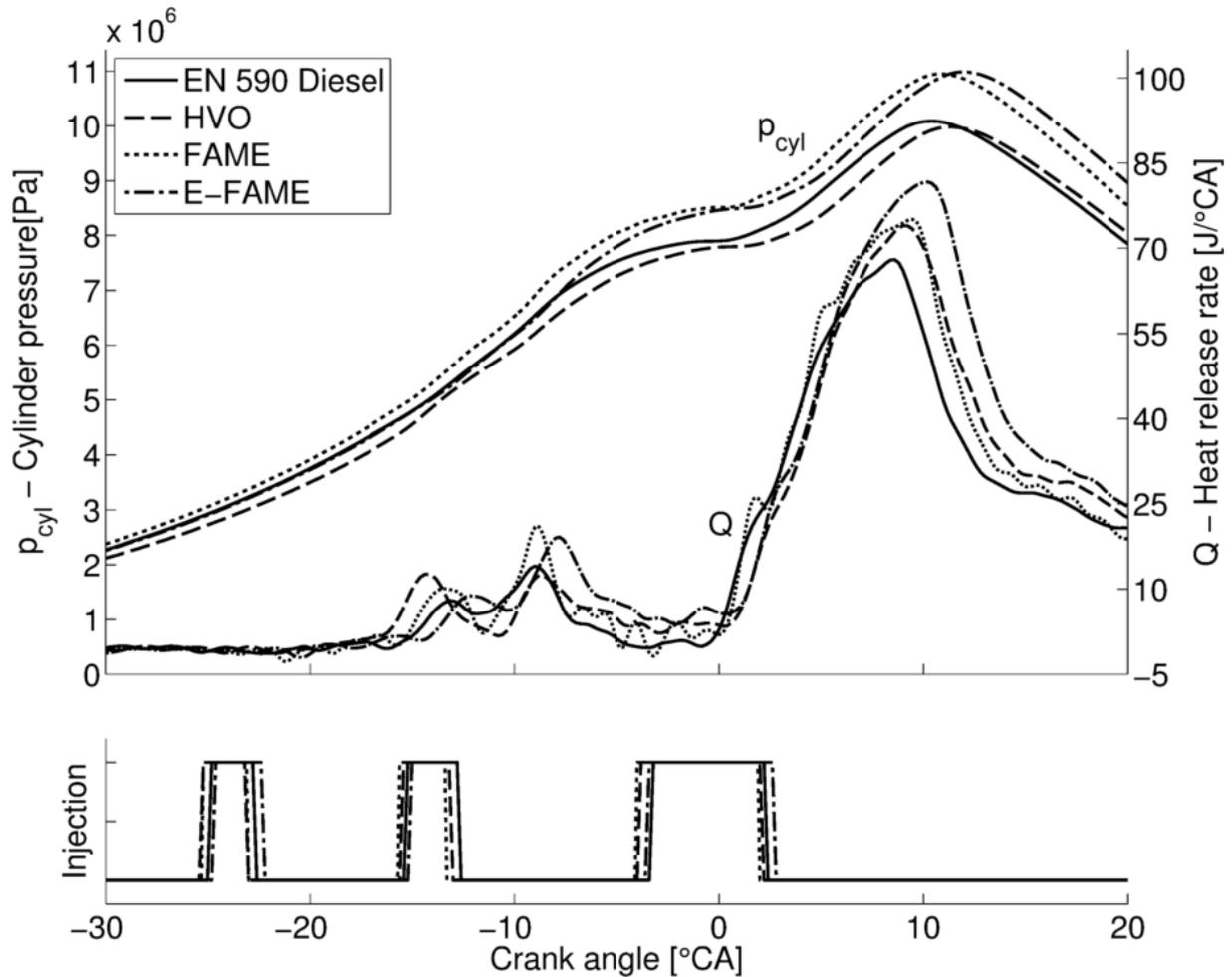


Figure 9: Cylinder pressures (p_{cyl}), heat release rates (Q) and injection signals for EN 590 diesel, HVO, FAME and E-FAME; $n = 2,000 \text{ min}^{-1}$, $M = 100 \text{ Nm}$

Operation with FAME also shows a slight shift towards an earlier combustion of the first fuel injection which is reasoned by its slightly higher cetane number compared to EN 590 diesel. Start of second pre-combustion and main combustion is not shifted. However, heat release rates are increased and the duration of the main combustion is prolonged compared to EN 590 diesel caused by a slightly increased main injection. Furthermore a very minor premix-peak is apparent. For the operation with HVO, E-FAME and EN 590 diesel no premix-peak is apparent. Caused by its decreased cetane number E-FAME shows a slightly delayed start of combustion in relation to the pre-combustions as well as the main combustion. Duration and maximum heat release of the main combustion with E-FAME are significantly increased compared to EN 590 diesel also due to a slightly increased main injection.

Caused by the decreased heating values of FAME and E-FAME operation points are shifted in the engine maps of the ECU. In order to get the same BMEP higher injection rates are needed. The amount of injected fuel influences important control parameters

like charge air pressure and rail pressure, Figure 10. The level of the cylinder pressure of FAME in Figure 9 is higher than that of EN 590 diesel for the complete operation cycle. Moreover, the positions of injections are also influenced by the shift of operation points in the engine maps.

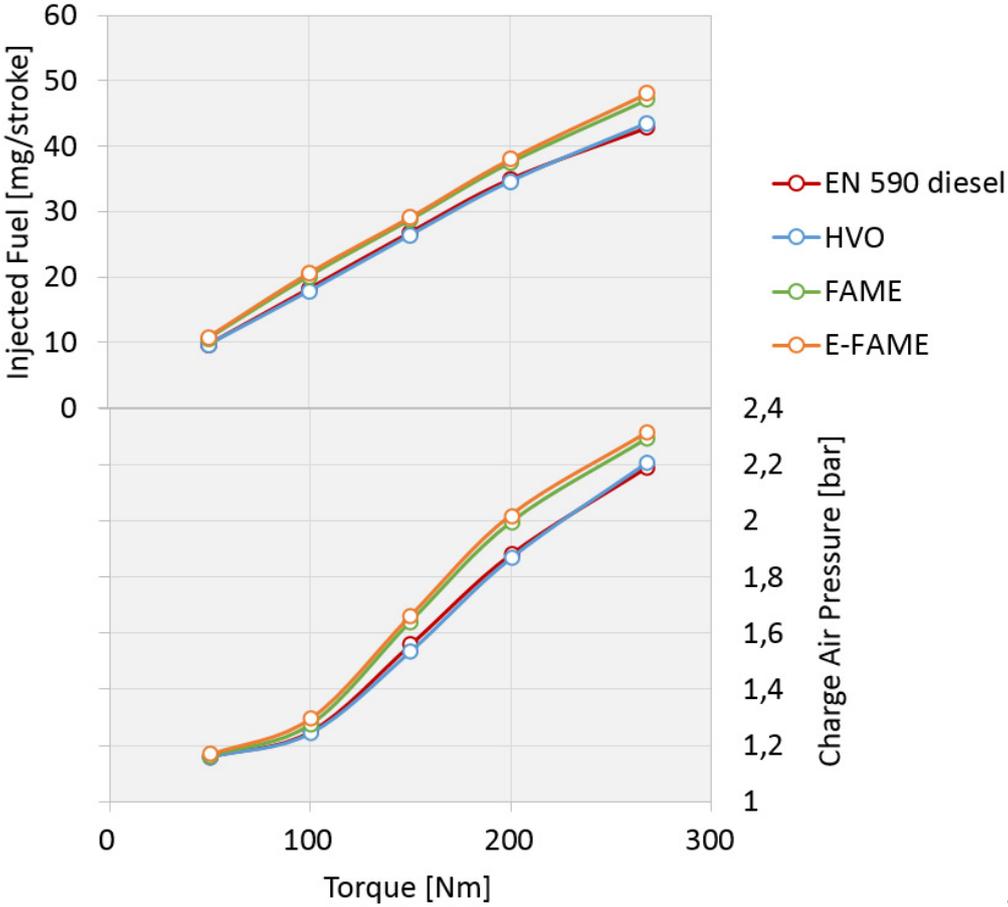


Figure 10: Total amount of injected fuel (upper graph) and charge air pressure (lower graph) for EN 590 diesel, HVO, FAME and E-FAME at $n = 2,000 \text{ min}^{-1}$

7.2.2. Impacts on engine control parameters

The impact of the investigated biofuels is highly depend on the engine operation. Figure 11 shows the absolute change of EGR-rate for HVO, FAME and E-FAME within the engine map. It is apparent that the changes for HVO are negligible while the operation with FAME shows a slight shift towards lower EGR-rates in low load conditions. The same trend is visible for E-FAME operation though clearly more distinct. Especially at low speeds and medium torques up to 150 Nm E-FAME shows a significant impact towards decreased EGR-rates. Exhaust gas recirculation is used in order to lower combustion temperatures and thus decreasing NOx emissions. A decreased EGR-rate will therefore lead to higher NOx emissions and decreased soot emissions in the raw exhaust gas. It should be noted that less or even no exhaust gas

recirculation is applied towards higher loads and speeds. Therefore, the absolute impacts on the EGR-rate are less distinct in these parts of the engine map.

Figure 12 shows the impact of the investigated biofuels on the fuel rail pressure within the engine map. Again, HVO operation shows negligible shifts of the fuel rail pressure since the heating value of HVO is similar to EN 590 diesel and therefore ECU control parameters are less affected. FAME operation shows significant increase of the rail pressure at speeds above 1500 min^{-1} and low loads. Another shift towards higher rail pressures is visible at high speed and high load conditions. The impact of E-FAME is similar though more distinct as when operating with FAME. Increased rail pressures lead to increased combustion temperatures which result in higher NO_x emissions and lower soot emissions.

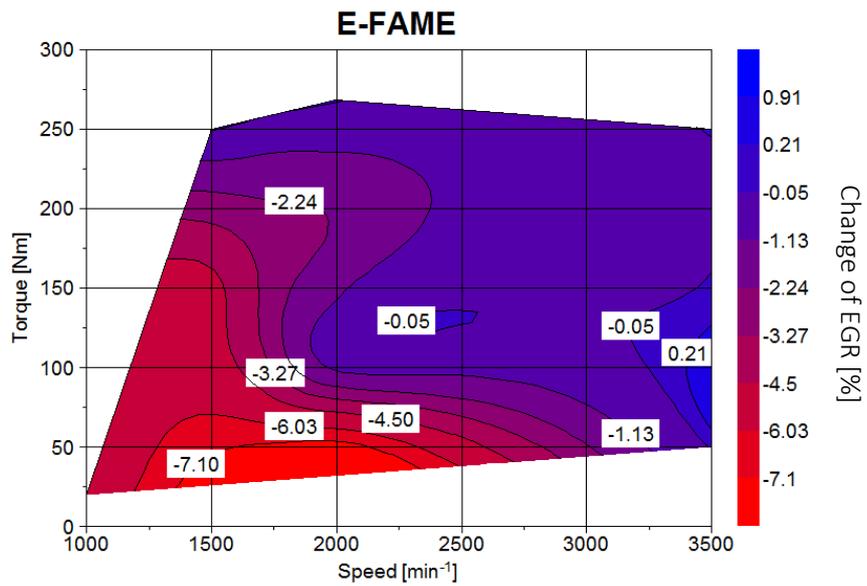
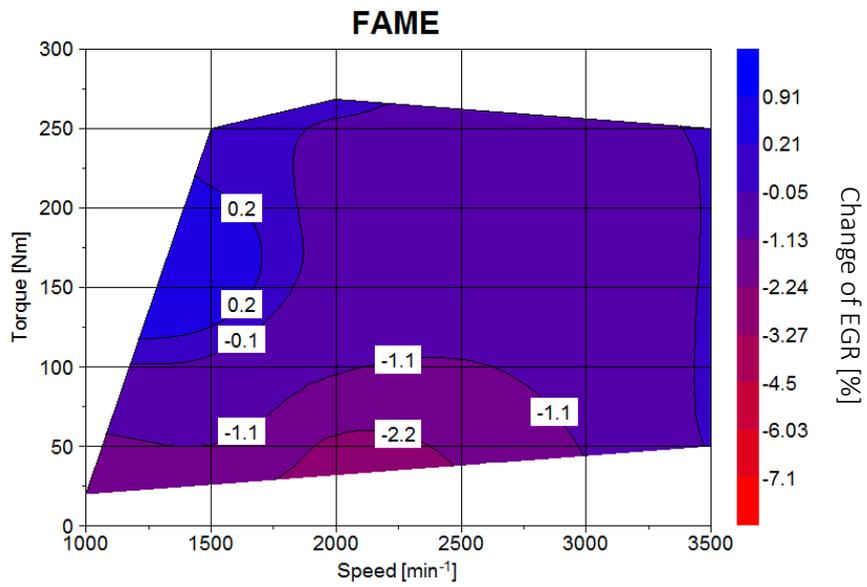
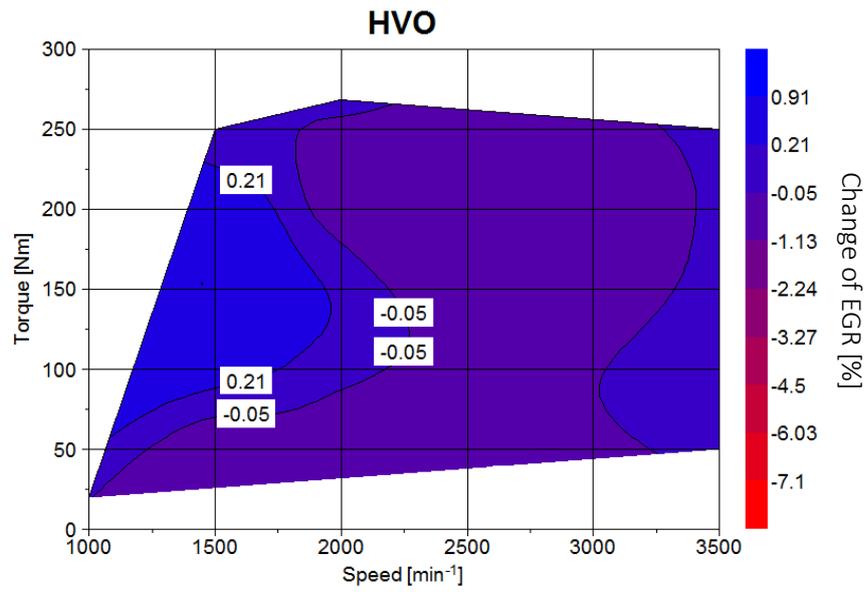


Figure 11: Absolute change of EGR-rate through HVO (upper graph), FAME (middle gr.) and E-FAME (lower gr.)

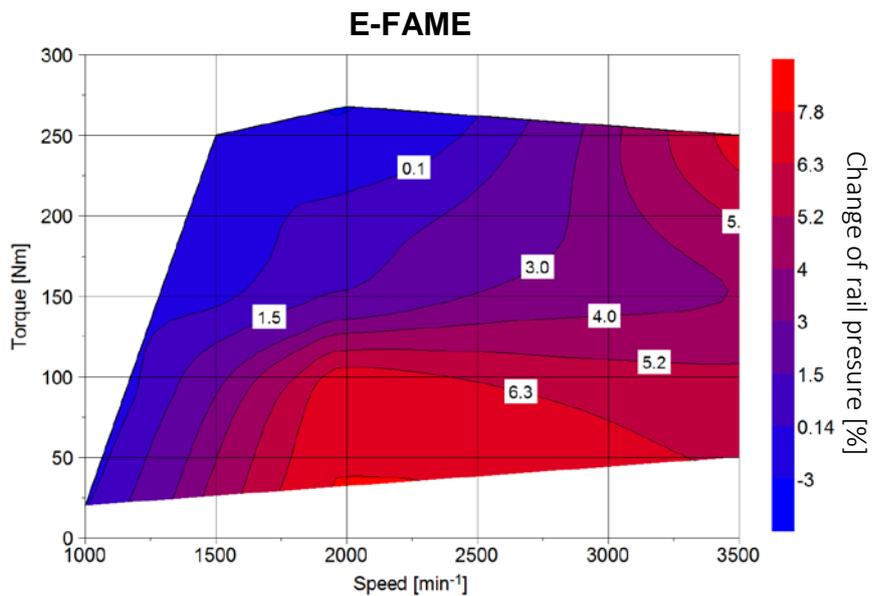
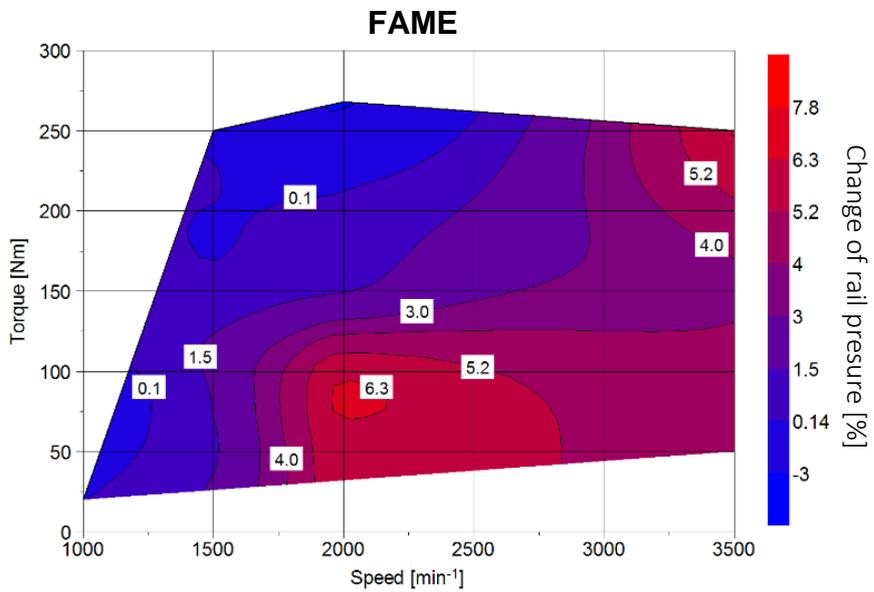
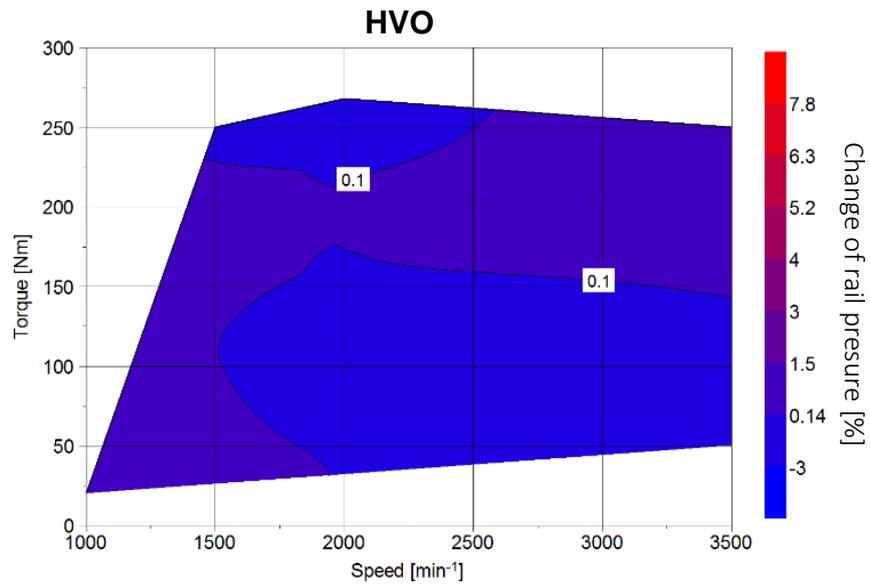


Figure 12: Rel. change of rail pressure through HVO (upper graph), FAME (middle gr.) and E-FAME (lower gr.)

7.2.3. Impacts on emissions

The analysis of raw exhaust emissions shows clear influences of the biofuels (Figure 13). The emissions shown are based on averaged and non-weighted values measured at the selected operations points shown in Figure 6. For HVO operation a significant reduction of soot emissions is apparent by a decreased FSN value. The decrease in soot emission is likely caused by the absence of aromatic and polyaromatic compounds in the fuel [1]. Moreover, the decreased final boiling point of HVO compared to EN 590 diesel positively affects the mixture formation and thus decreases the emission of unburned or partly burned hydrocarbons. This can also be seen in slightly decreased emissions of CO and THC. However, emissions of NO_x are on a similar level as for EN 590 diesel.

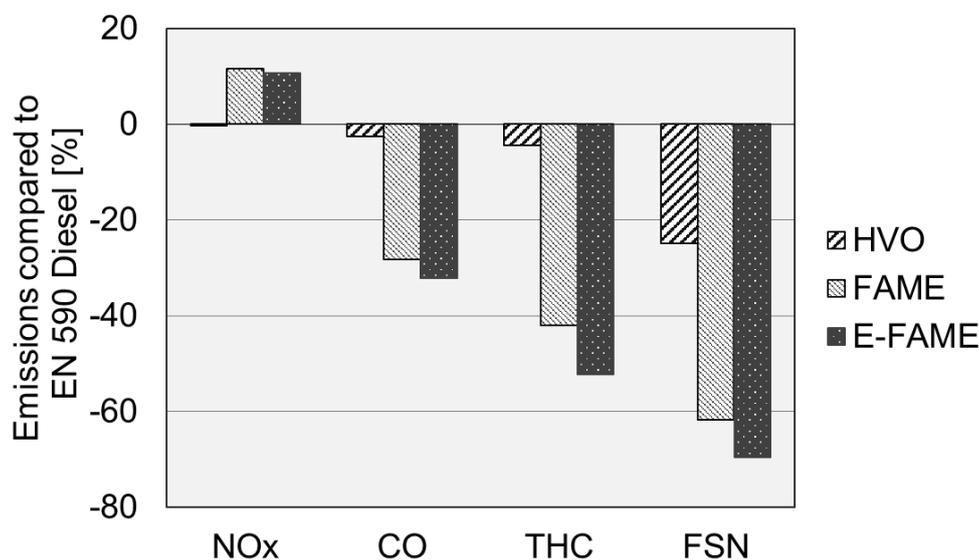


Figure 13: Average, non-weighted raw emissions compared to EN 590 diesel

Operation with FAME and E-FAME leads to a more complete combustion caused by the oxygen content in both fuels. This results in clearly reduced emissions of soot, CO and THC compared to EN 590 diesel and HVO. The additional fuel bound oxygen reduces local zones of deficient air during mixture formation. Furthermore, the oxygen improves soot oxidation processes. Slightly increased NO_x emissions are apparent for FAME and E-FAME in contrast to HVO operation. Increased NO_x emissions during FAME and E-FAME operation are to some extent based on the decreased EGR-rates. Emission performance of E-FAME is slightly better than that of FAME which may be caused by a higher oxygen content or the increased impacts on ECU control parameters as shown in Figure 11 and Figure 12. A significant influence of the biofuels on the tailpipe emissions could not be determined.

The impacts on raw exhaust emissions are depend on the operation point within the engine map, Figure 14. It is apparent that the decrease of soot emissions through HVO mainly occurs at low speed conditions where exhaust gas recirculation is applied. The impacts on EGR-rate and rail pressure during HVO operation are negligible as shown in Figure 11 and Figure 12. Consequentially, the reduction of soot is mainly affected by the fuel properties in this case. The distribution of soot benefits within the engine map is similar for FAME and E-FAME. Soot emissions are mostly reduced at speeds below $2,500 \text{ min}^{-1}$. The decreased EGR-rate for E-FAME at low speeds likely supports low soot behavior in this region of the engine map. It can be stated that the reduced soot emissions for HVO, FAME and E-FAME allow an extended use of exhaust gas recirculation in order to further reduce NO_x emissions.

Figure 15 shows the change of NO_x emissions for HVO, FAME and E-FAME within the engine map compared to operation with EN 590 diesel. While with HVO nearly no impact on NO_x emissions for the whole engine map are apparent, clear differences can be seen for FAME and E-FAME operation. Especially at high speed conditions NO_x emissions are significantly increased compared to operation with EN 590 diesel. These zones in the engine map are characterized by a non-applied exhaust gas recirculation. That means, that most disadvantages in NO_x emissions are not based on the shifted EGR-rates. However, rail pressures are increased at high speeds which also results in increased NO_x emissions.

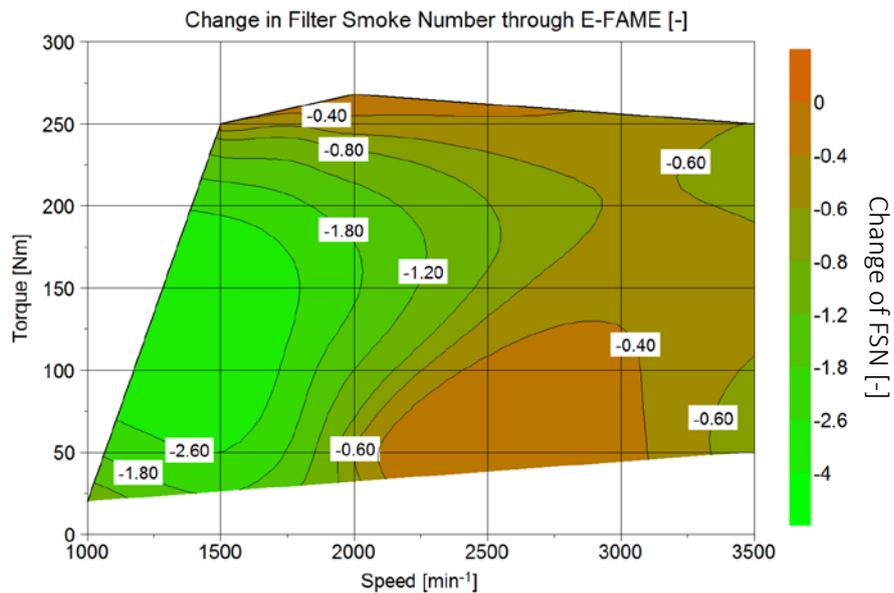
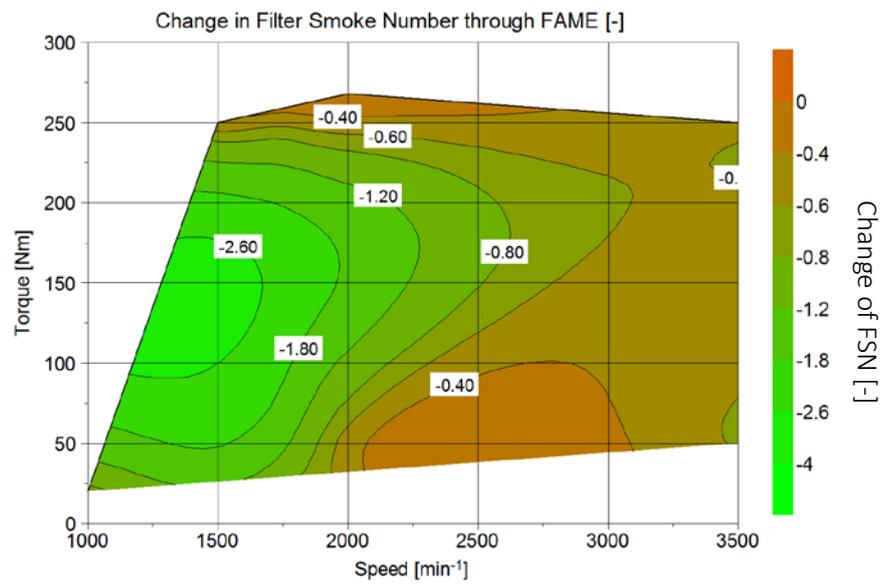
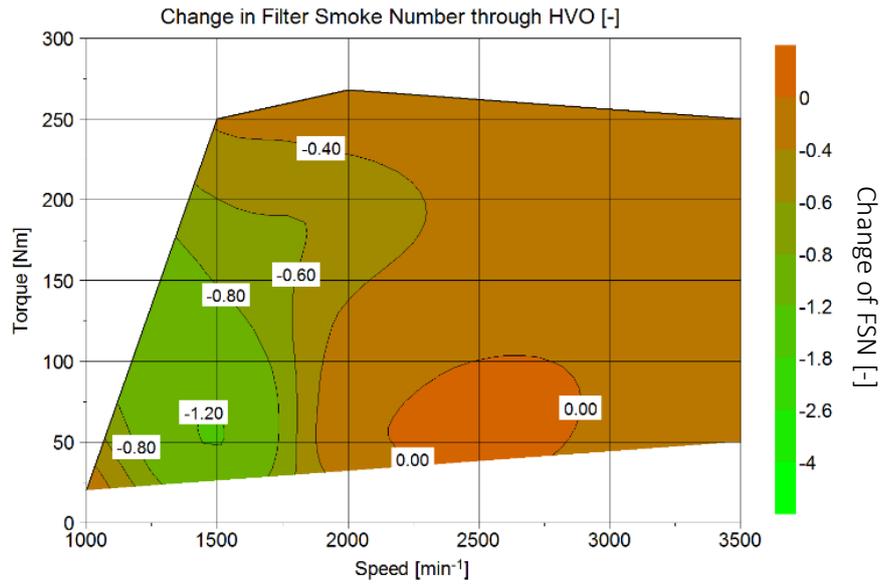


Figure 14: Change in raw soot emissions through HVO (upper graph), FAME (middle gr.) and E-FAME (lower gr.)

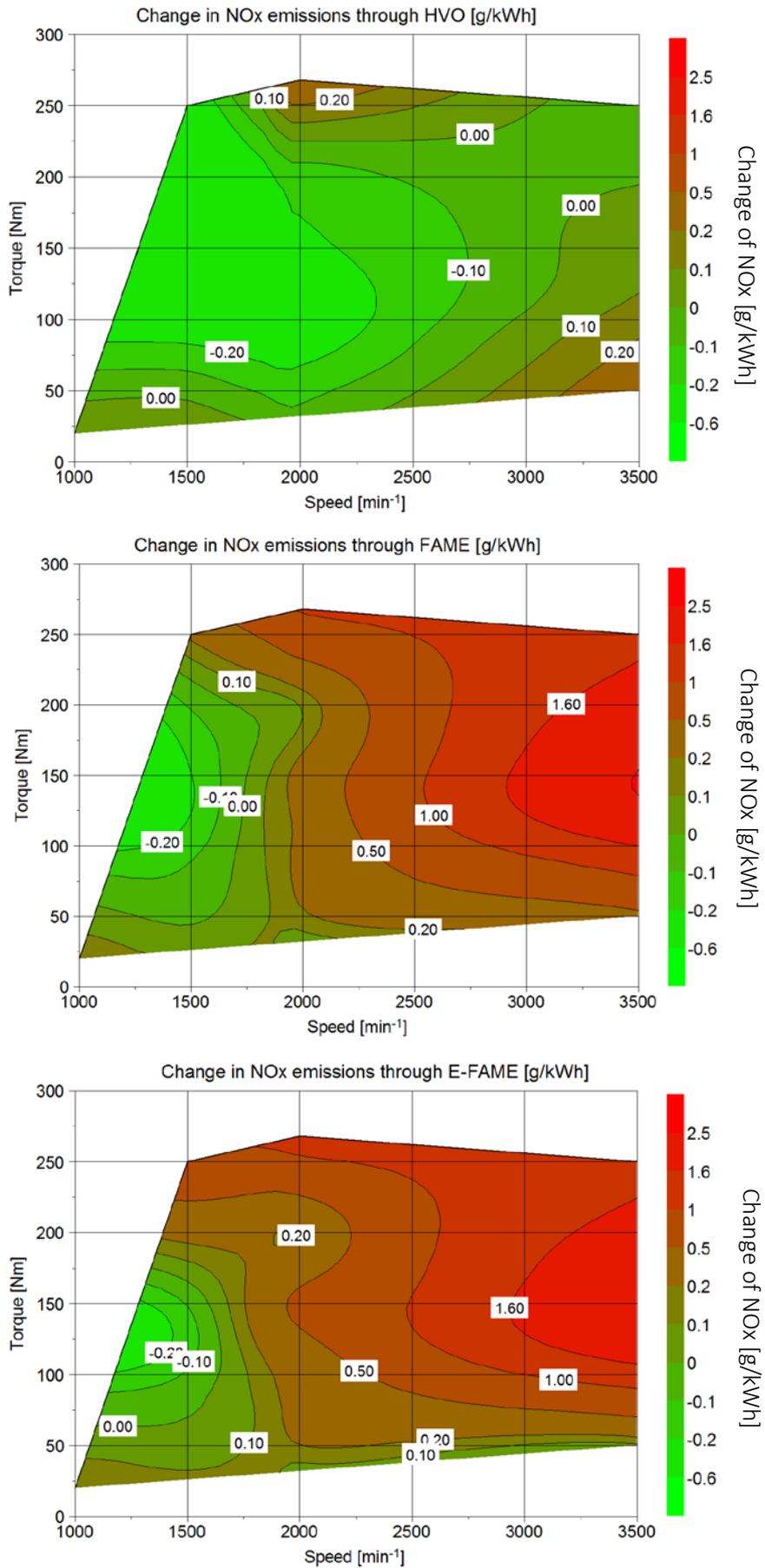


Figure 15: Change in raw NOx emissions through HVO (upper graph), FAME (middle gr.) and E-FAME (lower gr.)

7.3. Open ECU operation

Chapter 7.2.2 showed that shifts in ECU control parameters occurred for the investigated biofuels due to their fuel properties. In order to achieve the best performance for HVO and E-FAME a fuel adapted ECU calibration is needed. The ECU contains various engine maps for control parameters like EGR-throttle position or rail pressure. It was possible to adapt the control settings for HVO and E-FAME using the application tool IAV FI^{2RE}, Figure 16. This open ECU replaced the original ECU for these experiments and was able to operate the engine with control of:

- Injector current feed (multiple injections possible)
- Throttle actuator
- EGR actuator
- Charging valve for high-pressure fuel pump control
- Rail pressure control valve
- VTG actuator.

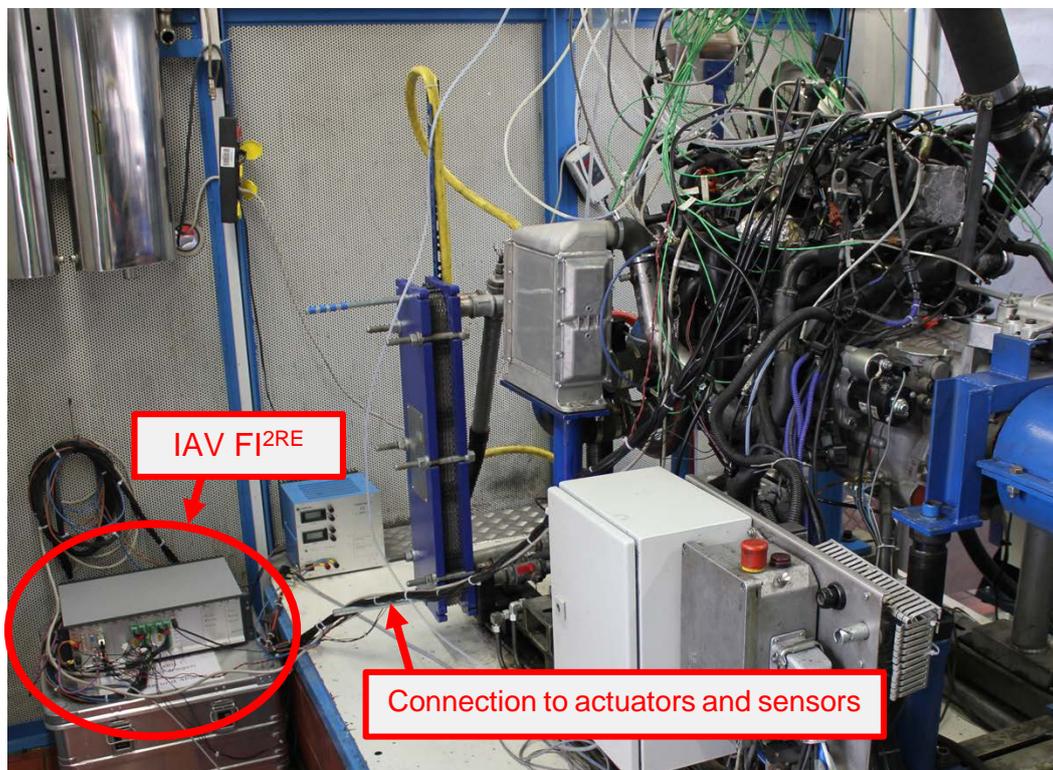


Figure 16: Open ECU setup

However, since the control strategies of the original ECU are very complex and not fully accessible the open ECU can only partly reproduce an engine operation with

original settings. Therefore all fuels were operated and measured again using ECU settings (optimized for EN 590 diesel) which were recorded before from the original ECU. Subsequently the control settings were optimized for HVO and E-FAME. The goal of the optimization process was to minimize biofuel consumption while still meeting current emission limits for diesel operation. The method to achieve this goal is illustrated in Figure 17.

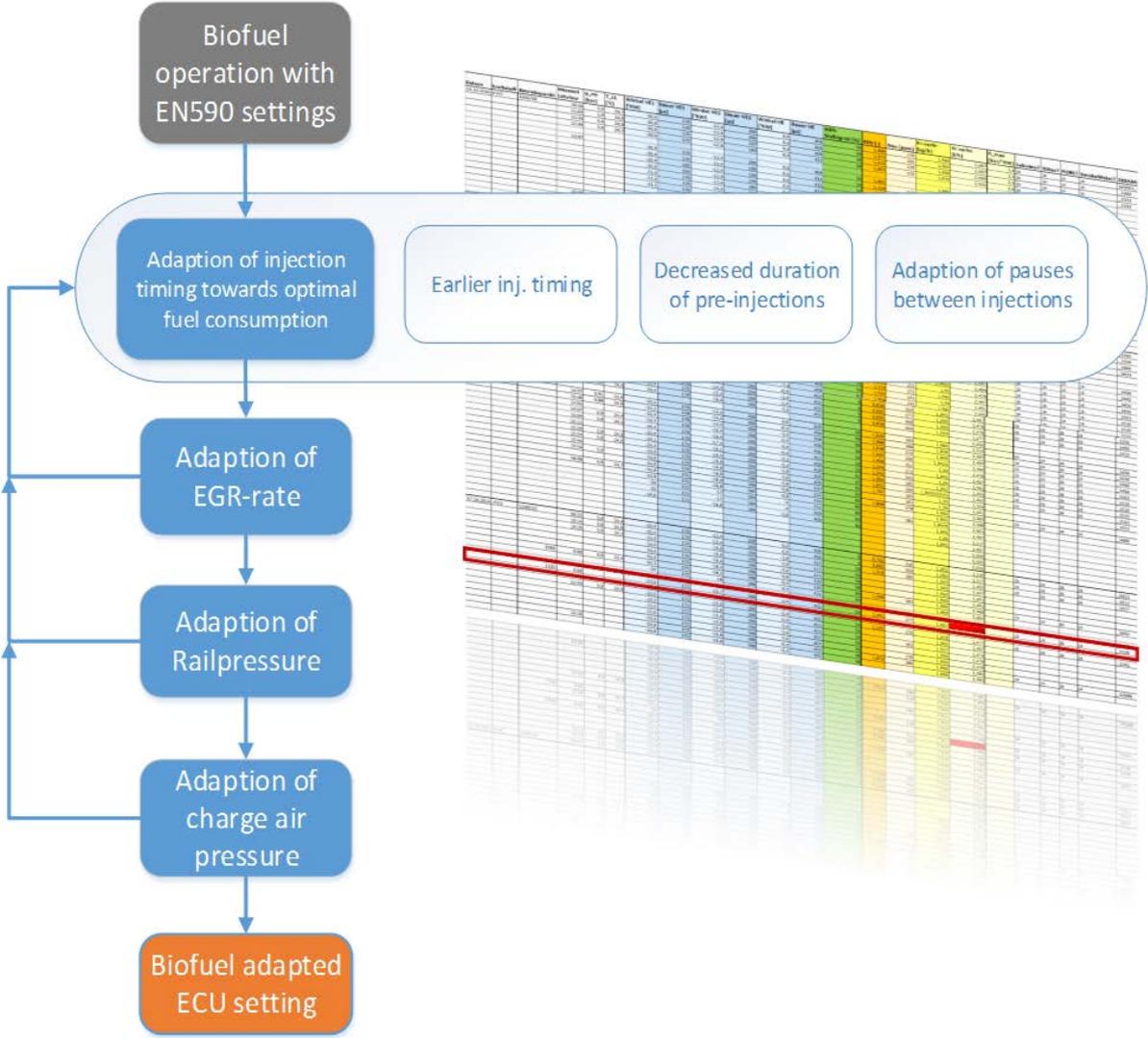


Figure 17: Method of adapting ECU settings for biofuel operation

The optimization process is an iterative process that adapts the injection settings, EGR-rate, rail pressure and charge air pressure. The optimization of the injection was done through adapting the injection timing, the pauses between multiple injections and the duration of the pre-injections, Figure 18.

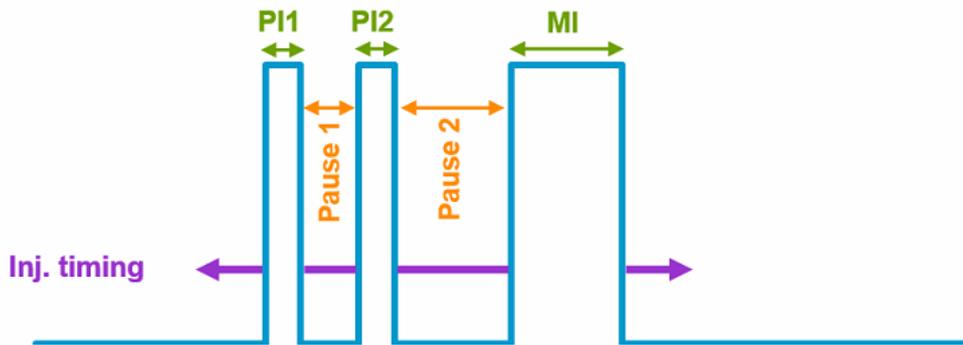


Figure 18: Adaption of injection settings (PI: pre-injection; MI: main injection)

7.3.1. Impacts on fuel efficiency and emissions

Figure 19 shows the fuel consumption of HVO and E-FAME at two selected operation points in comparison to standard EN 590 diesel. The second operation point ($n = 3,500 \text{ min}^{-1}$, $M = 150 \text{ Nm}$) is characterized as an operation point with high speed and high load that is not relevant for European emission certification which uses the New European Driving Cycle (NEDC). Therefore engines are rather optimized for power output respectively engine protection at high speeds and high loads.

It is apparent from Figure 19 that the mass based fuel consumption of HVO is below the consumption of diesel which is reasoned by the higher heating value of HVO. However, the operation with an ECU setting optimized for HVO shows that the fuel consumption could be reduced even further at both operation points. NO_x emissions of HVO meet the limits at $n = 2,000 \text{ min}^{-1}$, $M = 100 \text{ Nm}$ (Figure 20). At $n = 3,500 \text{ min}^{-1}$, $M = 150 \text{ Nm}$ NO_x emissions are higher than for EN 590 diesel, though this operation point is not relevant for emission certification. Soot emissions are still below diesel levels at both operation points.

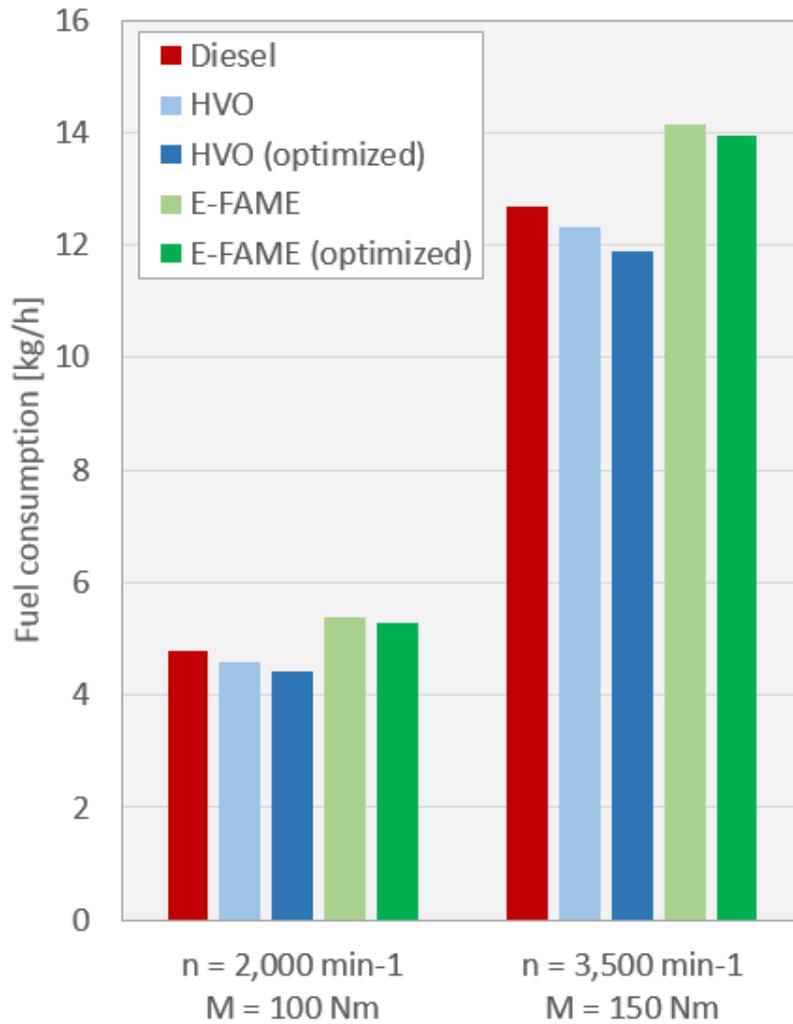


Figure 19: Fuel consumption of EN 590 diesel, HVO and E-FAME at selected operation points

The operation with E-FAME shows slightly higher fuel consumptions since its heating value is rather low compared to EN 590 diesel and HVO. However, an E-FAME adapted ECU setting could still improve fuel efficiency. Again, NO_x and soot emissions still meet the limit at $n = 2,000 \text{ min}^{-1}$, $M = 100 \text{ Nm}$. At $n = 3,500 \text{ min}^{-1}$, $M = 150 \text{ Nm}$ NO_x emissions are above EN 590 diesel levels though soot emissions are still below.

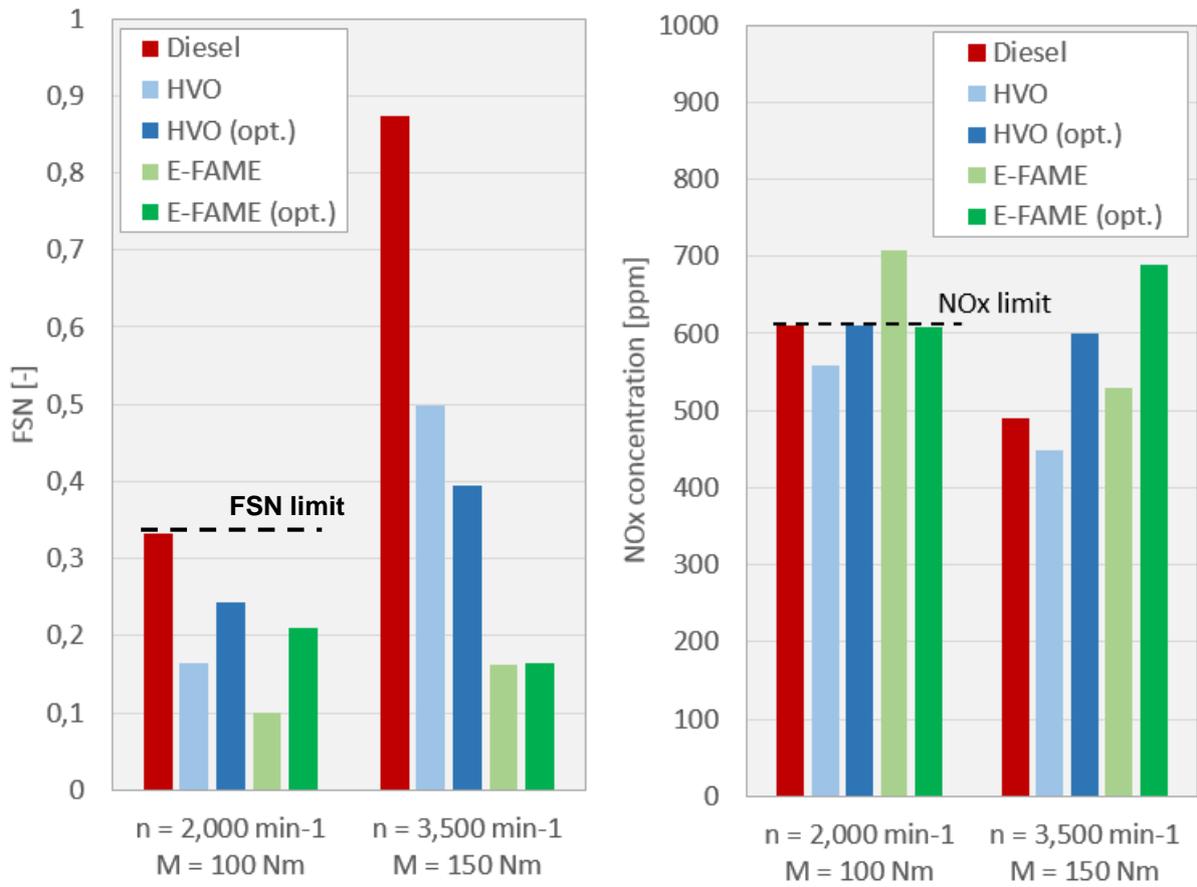


Figure 20: Left: Filter smoke number (FSN), right: NOx concentration in raw exhaust gas for EN 590 diesel, HVO and E-FAME at selected operation points

8. Conclusion

Comparing the different fuels, it is clear that HVO is a product of higher value than FAME/E-FAME. This is mainly because of the superior properties of HVO over FAME/E-FAME; low density, high cetane number, no inherent blending limit. HVO is less energy consuming than FAME/E-FAME during production. This is due to the simpler process of HVO and the use of excess propanol in the plant. The greenhouse gas emissions from the production of the fuels are in the same proximity; however, all of the alternative diesels investigated here have a lower outlet of greenhouse gases during production than standard diesel. The hydrotreating into HVO might be a more expensive process than transesterification into FAME, because no byproducts are generated.

The choice between making HVO and FAME might be a question of demand and available secondary feedstock supply. If FAME is the chosen product, enzymes as catalyst might be worth investigating further, especially with 2nd generation biodiesel with low cost/low quality feedstock with a high content of FFA.

Engine tests revealed that HVO positively affects the combustion and raw emissions even if standard ECU settings are used. Moreover, HVO-adapted ECU settings showed a decreased fuel consumption without exceeding current emission limits. Based on the performed engine tests HVO can be characterized as very suitable for modern diesel engines.

Using FAME or E-FAME as a diesel substitute will enormously decrease the formation of partly burned combustion products and soot in raw exhaust, though, NO_x emissions may be increased. E-FAME adapted ECU settings could reduce NO_x emissions to the same level as for diesel and decrease fuel consumption compared to standard ECU operation. The engine tests showed that an engine operation with FAME could benefit from strongly reduced raw emissions, however, disadvantages of FAME regarding long-term stability and material incompatibility need to be considered as well.

9. List of Figures

Figure 1: Evolution of European emission standards for passenger car diesel engines, [3]	2
Figure 2: Schematic of different routes to biofuels. The specific fuel and fuel routes tested and analyzed in this project is highlighted in yellow	3
Figure 3: Overall properties of FAME biodiesel depending on source of bio oil.....	4
Figure 4: The quality index is based on cetane number, storage stability and CFPP (Cold Filter Plugging Point) of the fuels, the cost index is based on different sources and estimations	6
Figure 5: Overview of energy use and GHG emissions during the production of 1MJ fuel (LHV)	11
Figure 6: Selected operation points for fuel testing.....	13
Figure 7: Scheme of the test engine.....	15
Figure 8: Test engine with exhaust after treatment components and test bench brake	16
Figure 9: Cylinder pressures (p_{cyl}), heat release rates (Q) and injection signals for EN 590 diesel, HVO, FAME and E-FAME; $n = 2,000 \text{ min}^{-1}$, $M = 100 \text{ Nm}$	17
Figure 10: Total amount of injected fuel (upper graph) and charge air pressure (lower graph) for EN 590 diesel, HVO, FAME and E-FAME at $n = 2,000 \text{ min}^{-1}$	18
Figure 11: Absolute change of EGR-rate through HVO (upper graph), FAME (middle gr.) and E-FAME (lower gr.).....	20
Figure 12: Rel. change of rail pressure through HVO (upper graph), FAME (middle gr.) and E-FAME (lower gr.).....	21
Figure 13: Average, non-weighted raw emissions compared to EN 590 diesel.....	22
Figure 14: Change in raw soot emissions through HVO (upper graph), FAME (middle gr.) and E-FAME (lower gr.).....	24
Figure 15: Change in raw NOx emissions through HVO (upper graph), FAME (middle gr.) and E-FAME (lower gr.).....	25
Figure 16: Open ECU setup	26
Figure 17: Method of adapting ECU settings for biofuel operation	27
Figure 18: Adaption of injection settings (PI: pre-injection; MI: main injection).....	28
Figure 19: Fuel consumption of EN 590 diesel, HVO and E-FAME at selected operation points.....	29

Figure 20: Left: Filter smoke number (FSN), right: NOx concentration in raw exhaust gas for EN 590 diesel, HVO and E-FAME at selected operation points 30

10. References

- [1] David Appleyard, Green replacement fuels taking flight, *Renewable Energy Focus*, Volume 15, Issue 1, January–February 2014, Pages 38-39, ISSN 1755-0084
- [2] Sadlowski, T.; Richter, B.; Wichmann, V.; Harndorf, H. (2013): Storage Stability and Performance of Biofuel Blends, *MTZ worldwide* 74 (7-8), 56-61.
- [3] Wichmann, V.; Sadlowski, T.; Stengel, B. (2013): Einsatz von rapsölbasierten Kraftstoffen in Dieselmotoren, 7. Rostock Bioenergieforum.
- [4] S. Mikkonen, T. Hartikka, M. Kuronen, P. Saikkonen, »HVO, Hydrotreated Vegetable Oil - A premium renewable biofuel for diesel engines« Neste Oil Proprietary publication, 2012.
- [5] R. Edwards, J.-F. Larivé, D. Rickeard og W. Weindorf, »Well-to-Tank Report Version 4.a JEC Well-to-Wheels Analysis« Joint Research center, 2014.
- [6] Frenklach, M. (2002): Reaction Mechanism of Soot Formation in Flames, *Physical Chemistry Chemical Physics* (11), 2028-2037.
- [7] L. F. Sotoft, B.-G. Rong, K. V. Christensen og B. Norddahl, »Process simulation and economical evaluation of enzymatic biodiesel production plant« *Elsevier*, 27 November 2009.

Appendix

A Report of fuel analysis (standard diesel EN 590, HVO, E-FAME)

Alberta Innovates - Technology Futures ~ Fuels & Lubricants



250 Karl Clark Road, Edmonton, Alberta, Canada T6N 1E4
 Certified by the Standards Council of Canada as an Accredited Testing
 Organization complying with the requirements of ISO/IEC 17025 for
 specific tests registered with the Council



Report of Analysis

This report may only be reproduced in its entirety

Reported: 19-Sep-2014
 Revision: 2014-1

Order Id: FL14_0844
 Contract #:
 PO#: 3000548913

Report To:

CanmetENERGY
 1 Oil Patch Drive Suite A202
 Devon, AB T9G 1A8

Attention: Craig Fairbridge
 E-mail: Craig.Fairbridge@NRCan-RNCan.gc.ca
 Fax: 780 987 8676

Invoice To:

CanmetENERGY
 1 Oil Patch Drive Suite A202
 Devon, AB T9G 1A8

Attention: Craig Fairbridge
 E-mail:
 Fax:

Laboratory Sample Number: FL14_0844-001
 Product: Diesel
 Specification: EN 590:2013
 Date Received: 12-Jun-2014

Sample Source
 Reference: Reference Diesel
 Location:
 Tag Number:
 Tank Number:

Sample Notes
 1

Specification Details

Analysis	Test Name	Specifications		Results	Units	Test Notes
		Minimum	Maximum			
Copper Corrosion - Classification	ASTM D130	Class 1	Class 1	1a		
Total Insolubles, g/m3	ASTM D2274		25	1.1	g/m3	
Density @ 15°C	ASTM D4052	820.0	845.0	838.8	kg/m3	
Kinematic Viscosity	ASTM D445 @ 40°C	2.000	4.500	2.714	mm2/s (cSt)	
Carbon Residue, 10% Bottoms	ASTM D4530		0.30	0.01	Mass %	
Calculated Cetane Index, Procedure B	ASTM D4737	46.0		50.2		
Ash Content	ASTM D482		0.010	0.005	Mass %	
Particulate Contamination	ASTM D5452		24 (mg/kg)	0.84	mg/L	
Total Sulfur	ASTM D5453		10.0	7.6	mg/kg	
Cloud Point	ASTM D5773	Report	Report	-6.1	°C	
Cetane Number	ASTM D613	51.0		53.1		
Water Content	ASTM D6304		200	55	mg/kg	
Manganese (Mn)	ASTM D7111		2.0 (mg/L)	<0.01	mg/kg	
Biodiesel Content	ASTM D7371		7.0	6.35	Volume %	
Wear Scar Diameter	ASTM D7688		460	220	um	
Distillation 95% Recovered (corr)	ASTM D86		360	347.7	°C	
% Recovered at 250°C	ASTM D86		<65	37	% (v/v)	
% Recovered at 350°C	ASTM D86	85		96	% (v/v)	
Corrected Flash Point	ASTM D93	>55.0		63.5	°C	
Oxidation Stability @ 110°C	EN 15751	20		12.3	hours	
CFPP	SSWRI - ASTM D6371	Report	Report	-27	°C	2

Tested Parameters (Note: Parameters in Specification Detail will also appear in complete listing)

Analysis	Test Name	Results	Units	Test Notes
Copper Corrosion - Test Duration	ASTM D130	3	hours	
Copper Corrosion - Test Temperature	ASTM D130	50	°C	
Copper Corrosion - Classification	ASTM D130	1a		
Total Insolubles, g/m3	ASTM D2274	1.1	g/m3	
Density @ 15°C	ASTM D4052	838.8	kg/m3	
Kinematic Viscosity	ASTM D445 @ 40°C	2.714	mm2/s (cSt)	
Carbon Residue, 10% Bottoms	ASTM D4530	0.01	Mass %	
Calculated Cetane Index, Procedure B	ASTM D4737	50.2		
Ash Content	ASTM D482	0.005	Mass %	
Volume of Fuel	ASTM D5452	1000	mL	
Particulate Contamination	ASTM D5452	0.84	mg/L	
Total Sulfur	ASTM D5453	7.6	mg/kg	

Alberta Innovates - Technology Futures ~ Fuels & Lubricants

250 Karl Clark Road, Edmonton, Alberta, Canada T6N 1E4

Certified by the Standards Council of Canada as an Accredited Testing
Organization complying with the requirements of ISO/IEC 17025 for
specific tests registered with the Council



Report of Analysis

This report may only be reproduced in its entirety

Reported: 19-Sep-2014

Revision: 2014-1

Order Id: FL14_0844

Contract #:

PO#: 3000548913

Page 2 of 3

Tested Parameters (Note: Parameters in Specification Detail will also appear in complete listing)

Analysis	Test Name	Results	Units	Test Notes
Cloud Point	ASTM D5773	-6.1	°C	
Cetane Number	ASTM D613	53.1		
Water Content	ASTM D6304	55	mg/kg	
Manganese (Mn)	ASTM D7111	<0.01	mg/kg	
Biodiesel Content	ASTM D7371	6.35	Volume %	
Major Axis	ASTM D7688	0.24	mm	
Minor Axis	ASTM D7688	0.21	mm	
Wear Scar Diameter	ASTM D7688	220	um	
Distillation IBP	ASTM D86	168.5	°C	
Distillation 5% Recovered (corr)	ASTM D86	191.7	°C	
Distillation 10% Recovered (corr)	ASTM D86	203.1	°C	
Distillation 20% Recovered (corr)	ASTM D86	220.8	°C	
Distillation 30% Recovered (corr)	ASTM D86	238.5	°C	
Distillation 40% Recovered (corr)	ASTM D86	254.7	°C	
Distillation 50% Recovered (corr)	ASTM D86	270.4	°C	
Distillation 60% Recovered (corr)	ASTM D86	286.1	°C	
Distillation 70% Recovered (corr)	ASTM D86	301.4	°C	
Distillation 80% Recovered (corr)	ASTM D86	317.6	°C	
Distillation 90% Recovered (corr)	ASTM D86	334.5	°C	
Distillation 95% Recovered (corr)	ASTM D86	347.7	°C	
Distillation FBP	ASTM D86	357.7	°C	
Distillation Residue	ASTM D86	1.4	%	
% Recovered at 250°C	ASTM D86	37	% (v/v)	
Distillation Loss	ASTM D86	0.5	%	
% Recovered at 350°C	ASTM D86	96	% (v/v)	
Corrected Flash Point	ASTM D93	63.5	°C	
Oxidation Stability @ 110°C	EN 15751	12.3	hours	
CFPP	SSWRI - ASTM D6371	-27	°C	2

Alberta Innovates - Technology Futures ~ Fuels & Lubricants



250 Karl Clark Road, Edmonton, Alberta, Canada T6N 1E4
Certified by the Standards Council of Canada as an Accredited Testing
Organization complying with the requirements of ISO/IEC 17025 for
specific tests registered with the Council



Order Id: FL14_0844
Contract #:
PO#: 3000548913

Report of Analysis

This report may only be reproduced in its entirety

Reported: 19-Sep-2014
Revision: 2014-1

Page 3 of 3

Notes and Remarks

1. The sample was tested as per generally applicable requirement for automotive diesel fuel of European Standard Specification for Automotive Fuels - Diesel (EN 590:2013) by using equivalent ASTM methods. Cloud point and Cold Filter Plugging Point (CFPP) were performed as per climate-related requirements of the specification.
2. This work was subcontracted to a third party.

Approved by:

Linh Tran

Linh Tran

Instrumentation Analytical Coordinator

Results relate only to items tested.

Contact Information

Business Unit Manager: Dan Wispinski
Phone: (780) 450-5108
Email: dan.wispinski@albertainnovates.ca

Alberta Innovates - Technology Futures ~ Fuels & Lubricants



250 Karl Clark Road, Edmonton, Alberta, Canada T6N 1E4
 Certified by the Standards Council of Canada as an Accredited Testing
 Organization complying with the requirements of ISO/IEC 17025 for
 specific tests registered with the Council



Report of Analysis

This report may only be reproduced in its entirety

Reported: 17-Sep-2014
 Revision: 2014-1

Order Id: FL14_0844
 Contract #:
 PO#: 3000548913

Report To:

CanmetENERGY
 1 Oil Patch Drive Suite A202
 Devon, AB T9G 1A8

Attention: Craig Fairbridge
 E-mail: Craig.Fairbridge@NRCan-RNCan.gc.ca
 Fax: 780 987 8676

Invoice To:

CanmetENERGY
 1 Oil Patch Drive Suite A202
 Devon, AB T9G 1A8

Attention: Craig Fairbridge
 E-mail:
 Fax:

Laboratory Sample Number: FL14_0844-002
 Product: Diesel
 Specification: EN 590:2013
 Date Received: 12-Jun-2014

Sample Source
 Reference: HVO Neste Oil NEX BTL
 Location:
 Tag Number:
 Tank Number:

Sample Notes
 1

Specification Details

Analysis	Test Name	Specifications		Results	Units	Test Notes
		Minimum	Maximum			
Copper Corrosion - Classification	ASTM D130	Class 1	Class 1	1a		
Total Insolubles, g/m3	ASTM D2274		25	1.1	g/m3	
Density @ 15°C	ASTM D4052	820.0	845.0	778.8	kg/m3	
Kinematic Viscosity	ASTM D445 @ 40°C	2.000	4.500	2.868	mm2/s (cSt)	
Carbon Residue, 10% Bottoms	ASTM D4530		0.30	0.01	Mass %	
Calculated Cetane Index, Procedure B	ASTM D4737	46.0		78.3		
Ash Content	ASTM D482		0.010	0.003	Mass %	
Particulate Contamination	ASTM D5452		24 (mg/kg)	10.45	mg/L	
Total Sulfur	ASTM D5453		10.0	<1	mg/kg	
Cloud Point	ASTM D5773	Report	Report	-32.1	°C	
Cetane Number	ASTM D613	51.0		74.7		
Water Content	ASTM D6304		200	20	mg/kg	
Manganese (Mn)	ASTM D7111		2.0 (mg/L)	<0.01	mg/kg	
Biodiesel Content	ASTM D7371		7.0	<1.00	Volume %	2
Wear Scar Diameter	ASTM D7688		460	400	um	
Distillation 95% Recovered (corr)	ASTM D86		360	293.6	°C	
% Recovered at 250°C	ASTM D86		<65	8	% (v/v)	
% Recovered at 350°C	ASTM D86	85		>97	% (v/v)	
Corrected Flash Point	ASTM D93	>55.0		78.0	°C	
CFPP	SSWRI - ASTM D6371	Report	Report	-42	°C	3

Tested Parameters (Note: Parameters in Specification Detail will also appear in complete listing)

Analysis	Test Name	Results	Units	Test Notes
Copper Corrosion - Test Duration	ASTM D130	3	hours	
Copper Corrosion - Test Temperature	ASTM D130	50	°C	
Copper Corrosion - Classification	ASTM D130	1a		
Total Insolubles, g/m3	ASTM D2274	1.1	g/m3	
Density @ 15°C	ASTM D4052	778.8	kg/m3	
Kinematic Viscosity	ASTM D445 @ 40°C	2.868	mm2/s (cSt)	
Carbon Residue, 10% Bottoms	ASTM D4530	0.01	Mass %	
Calculated Cetane Index, Procedure B	ASTM D4737	78.3		
Ash Content	ASTM D482	0.003	Mass %	
Volume of Fuel	ASTM D5452	1000	mL	
Particulate Contamination	ASTM D5452	10.45	mg/L	
Total Sulfur	ASTM D5453	<1	mg/kg	
Cloud Point	ASTM D5773	-32.1	°C	

Alberta Innovates - Technology Futures ~ Fuels & Lubricants

250 Karl Clark Road, Edmonton, Alberta, Canada T6N 1E4

Certified by the Standards Council of Canada as an Accredited Testing
Organization complying with the requirements of ISO/IEC 17025 for
specific tests registered with the Council



Report of Analysis

This report may only be reproduced in its entirety

Reported: 17-Sep-2014

Revision: 2014-1

Order Id: FL14_0844

Contract #:

PO#: 3000548913

Page 2 of 3

Tested Parameters (Note: Parameters in Specification Detail will also appear in complete listing)

Analysis	Test Name	Results	Units	Test Notes
Cetane Number	ASTM D613	74.7		
Water Content	ASTM D6304	20	mg/kg	
Manganese (Mn)	ASTM D7111	<0.01	mg/kg	
Biodiesel Content	ASTM D7371	<1.00	Volume %	2
Major Axis	ASTM D7688	0.44	mm	
Minor Axis	ASTM D7688	0.36	mm	
Wear Scar Diameter	ASTM D7688	400	um	
Distillation IBP	ASTM D86	190	°C	
Distillation 5% Recovered (corr)	ASTM D86	239	°C	
Distillation 10% Recovered (corr)	ASTM D86	256.2	°C	
Distillation 20% Recovered (corr)	ASTM D86	267.6	°C	
Distillation 30% Recovered (corr)	ASTM D86	272.7	°C	
Distillation 40% Recovered (corr)	ASTM D86	275.9	°C	
Distillation 50% Recovered (corr)	ASTM D86	277.7	°C	
Distillation 60% Recovered (corr)	ASTM D86	279.9	°C	
Distillation 70% Recovered (corr)	ASTM D86	282.8	°C	
Distillation 80% Recovered (corr)	ASTM D86	285.6	°C	
Distillation 90% Recovered (corr)	ASTM D86	290.2	°C	
Distillation 95% Recovered (corr)	ASTM D86	293.6	°C	
Distillation FBP	ASTM D86	301.8	°C	
Distillation Residue	ASTM D86	1.3	%	
% Recovered at 250°C	ASTM D86	8	% (v/v)	
Distillation Loss	ASTM D86	0.5	%	
% Recovered at 350°C	ASTM D86	>97	% (v/v)	
Corrected Flash Point	ASTM D93	78.0	°C	
CFPP	SSWRI - ASTM D6371	-42	°C	3

Alberta Innovates - Technology Futures ~ Fuels & Lubricants



250 Karl Clark Road, Edmonton, Alberta, Canada T6N 1E4
Certified by the Standards Council of Canada as an Accredited Testing
Organization complying with the requirements of ISO/IEC 17025 for
specific tests registered with the Council



Order Id: FL14_0844
Contract #:
PO#: 3000548913

Report of Analysis

This report may only be reproduced in its entirety

Reported: 17-Sep-2014
Revision: 2014-1

Page 3 of 3

Notes and Remarks

1. The sample was tested as per generally applicable requirement for automotive diesel fuel of the European Standard Specification for Automotive Fuels - Diesel (EN 590:2013) by using equivalent ASTM methods. Cloud point and Cold Filter Plugging Point (CFPP) were performed as per climate-related requirements of the specification.
2. ASTM D7371 states "This test method covers the determination of the content of fatty acid methyl esters (FAME) biodiesel in diesel fuel oils. It is applicable to concentrations from 1.00 to 20 volume %.

The sample results has been reported as <1.00 volume %. However, the detection limit of FAME in diesel fuel has been determined to be <0.25 volume %. It is suspected that the actual FAME content in the sample is <0.25 volume %.

3. This work was subcontracted to a third party.

Approved by:

Linh Tran

Linh Tran

Instrumentation Analytical Coordinator

Results relate only to items tested.

Contact Information

Business Unit Manager: Dan Wispinski
Phone: (780) 450-5108
Email: dan.wispinski@albertainnovates.ca

Alberta Innovates - Technology Futures ~ Fuels & Lubricants



250 Karl Clark Road, Edmonton, Alberta, Canada T6N 1E4
 Certified by the Standards Council of Canada as an Accredited Testing
 Organization complying with the requirements of ISO/IEC 17025 for
 specific tests registered with the Council



Report of Analysis

This report may only be reproduced in its entirety

Reported: 17-Sep-2014
 Revision: 2014-1

Order Id: FL14_0844
 Contract #:
 PO#: 3000548913

Report To:

CanmetENERGY
 1 Oil Patch Drive Suite A202
 Devon, AB T9G 1A8

Attention: Craig Fairbridge
 E-mail: Craig.Fairbridge@NRCan-RNCan.gc.ca
 Fax: 780 987 8676

Invoice To:

CanmetENERGY
 1 Oil Patch Drive Suite A202
 Devon, AB T9G 1A8

Attention: Craig Fairbridge
 E-mail:
 Fax:

Laboratory Sample Number: FL14_0844-003

Product: Biodiesel

Specification: EN 14214:2012

Date Received: 12-Jun-2014

Sample Source

Reference: Enzymatisertes Fame, E-FAME

Location:

Tag Number:

Tank Number:

Sample Notes

1

Specification Details

Analysis	Test Name	Specifications		Results	Units	Test Notes
		Minimum	Maximum			
Copper Corrosion - Classification	ASTM D130	Class 1	Class 1	1a		
Density @ 15°C	ASTM D4052	860	900	882.3	kg/m3	
Kinematic Viscosity	ASTM D445 @ 40°C	3.50	5.00	4.245	mm2/s (cSt)	
Total Sulfur	ASTM D5453		10	2.5	mg/kg	
Cloud Point	ASTM D5773	Report	Report	-0.5	°C	
Cetane Number	ASTM D613	51.0		51.9		
Water Content	ASTM D6304		500	158	mg/kg	
Free Glycerin	ASTM D6584		0.02	<0.001	Mass %	
Total Glycerin	ASTM D6584		0.25	0.015	Mass %	
Total Monoglyceride	ASTM D6584		0.70	0.012	Mass %	
Total Diglyceride	ASTM D6584		0.20	<0.090	Mass %	
Total Triglyceride	ASTM D6584		0.20	0.001	Mass %	
Acid Number	ASTM D664		0.50	0.83	mg KOH/g	
Particulate Contamination	ASTM D7321		24 (mg/kg)	10.5	mg/L	
Ash Content	ASTM D874		0.02	<0.005	Mass %	
Corrected Flash Point	ASTM D93	101		141.0	°C	
Ester Content	EN 14103	96.5		95.3	% (m/m)	2
Linolenic Acid Methyl Ester Content	EN 14103		12.0	1.9	% (m/m)	2
Polyunsaturated Methyl Ester	EN 14103		1.00	<0.1	% (m/m)	2
Methanol Content	EN 14110		0.20	0.04	Mass %	
Iodine Value	EN 14111		120	116	g of I/100g	
Na and K, combined	EN 14538		5.0	<1.0	ppm (w/w)	3
Ca and Mg, combined	EN 14538		5.0	<1.0	ppm (w/w)	3
Phosphorus Content	EN 14538		4.0	<2.0	ppm (w/w)	3
Oxidation Stability @ 110°C	EN 15751	8.0		2.4	hours	
CFPP	SSWRI - ASTM D6371	Report	Report	-4	°C	

Tested Parameters (Note: Parameters in Specification Detail will also appear in complete listing)

Analysis	Test Name	Results	Units	Test Notes
Copper Corrosion - Test Duration	ASTM D130	3	hours	
Copper Corrosion - Test Temperature	ASTM D130	50	°C	
Copper Corrosion - Classification	ASTM D130	1a		
Density @ 15°C	ASTM D4052	882.3	kg/m3	
Kinematic Viscosity	ASTM D445 @ 40°C	4.245	mm2/s (cSt)	
Total Sulfur	ASTM D5453	2.5	mg/kg	
Cloud Point	ASTM D5773	-0.5	°C	

Alberta Innovates - Technology Futures ~ Fuels & Lubricants

250 Karl Clark Road, Edmonton, Alberta, Canada T6N 1E4

Certified by the Standards Council of Canada as an Accredited Testing
Organization complying with the requirements of ISO/IEC 17025 for
specific tests registered with the Council



Report of Analysis

This report may only be reproduced in its entirety

Reported: 17-Sep-2014

Revision: 2014-1

Order Id: FL14_0844

Contract #:

PO#: 3000548913

Page 2 of 3

Tested Parameters (Note: Parameters in Specification Detail will also appear in complete listing)

Analysis	Test Name	Results	Units	Test Notes
Cetane Number	ASTM D613	51.9		
Water Content	ASTM D6304	158	mg/kg	
Free Glycerin	ASTM D6584	<0.001	Mass %	
Total Glycerin	ASTM D6584	0.015	Mass %	
Total Monoglyceride	ASTM D6584	0.012	Mass %	
Total Diglyceride	ASTM D6584	<0.090	Mass %	
Total Triglyceride	ASTM D6584	0.001	Mass %	
Acid Number	ASTM D664	0.83	mg KOH/g	
Type of End Point	ASTM D664	Inflection		
Particulate Contamination	ASTM D7321	10.5	mg/L	
Sets of Filters Required	ASTM D7321	1	no unit	
Volume Filtered	ASTM D7321	0.4	L	
Ash Content	ASTM D874	<0.005	Mass %	
Corrected Flash Point	ASTM D93	141.0	°C	
Ester Content	EN 14103	95.3	% (m/m)	2
Linolenic Acid Methyl Ester Content	EN 14103	1.9	% (m/m)	2
Polyunsaturated Methyl Ester	EN 14103	<0.1	% (m/m)	2
Methanol Content	EN 14110	0.04	Mass %	
Iodine Value	EN 14111	116	g of I/100g	
Na and K, combined	EN 14538	<1.0	ppm (w/w)	3
Ca and Mg, combined	EN 14538	<1.0	ppm (w/w)	3
Phosphorus Content	EN 14538	<2.0	ppm (w/w)	3
Oxidation Stability @ 110°C	EN 15751	2.4	hours	
CFPP	SSWRI - ASTM D6371	-4	°C	

Alberta Innovates - Technology Futures ~ Fuels & Lubricants



250 Karl Clark Road, Edmonton, Alberta, Canada T6N 1E4
Certified by the Standards Council of Canada as an Accredited Testing
Organization complying with the requirements of ISO/IEC 17025 for
specific tests registered with the Council



Order Id: FL14_0844
Contract #:
PO#: 3000548913

Report of Analysis

This report may only be reproduced in its entirety

Reported: 17-Sep-2014
Revision: 2014-1

Page 3 of 3

Notes and Remarks

1. The sample was tested as per generally applicable requirement for biodiesel of the European Standard Specification for Fatty acid methyl esters (FAME) for use in diesel engines and heating applications (EN 14214:2012) by using equivalent ASTM methods. Cloud point and Cold Filter Plugging Point (CFPP) were performed as per climate-related requirements of the specification.
2. The EN 14103 method was modified to include the quantitation of polyunsaturated methyl esters (C20:4, C20:5, C22:5 and C22:6 FAMES).
3. The method was modified to include phosphorus. A Conostan S21+K multielement standard was used in place of the single element standard described in the method.

Approved by: _____

Linh Tran

Linh Tran

Instrumentation Analytical Coordinator

Results relate only to items tested.

Contact Information

Business Unit Manager: Dan Wispinski
Phone: (780) 450-5108
Email: dan.wispinski@albertainnovates.ca