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Diesel Emissions with DPF & SCR and Toxic Potentials with BioDiesel (RME) Blend Fuels

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ABSTRACT

The use of alternative fuels and among them the biofuels of 1st generation – fatty acid methyl esters FAME's *) and pure plants oils – for propulsion of IC engines is an important objective in several countries in order to save the fossil fuels and to limit the CO₂-production.

The properties of bio-fuels and bio-blend-fuels can vary and this has an impact on the operation and emissions of Diesel engines and on the modern exhaust aftertreatment systems.

The present paper represents the most important results obtained with RME at AFHB, EMPA and EC-JRC. Most of the activities were performed in the network project BioExDi (Biofuels, Exhaust Systems Diesel) in collaboration between industry and research institutes.

Following most important statements can be given about the treated subjects:

- emissions (engine-out) are influenced by RME in the sense less CO, HC & PM, more NO_x and more SOF,
- there are positive influences of RME on the functionality of DOC and DPF due to higher reactivity of RME, no influence on SCR,
- the long term functionality of exhaust aftertreatment systems with RME can be influenced by the potential higher availability of elements, like Ca, Mg, Zn, P, S, K,
- regarding particle mass PM: without DPF there are slight tendencies of lowering TEQ at low load with RME and increasing TEQ at high load of the engine; with DPF there is a strong reduction of all toxic substances and there are no effects of RME on PAH & TEQ.

Some practical recommendations for the users of biofuels of the 1st generation are given at the end of the paper. Generally there is a necessity of higher costs and efforts for more frequent service and maintenance and for the quality controls of fuel and fuel supply chain.

*) Abbreviations see at the end of this paper

INTRODUCTION AND PRELIMINARY INFORMATION

Application of RME in IC Engines

Rapeseed Oil Methyl Ester (RME), called also bio-diesel is one of many Fatty Acid Methyl Esters (FAME), which can be produced from different primary vegetable oils, like soya oil, palm oil, coconut oil, sunflower oil and others.

RME (FAME) can principally be used in all Diesel engines without changes of injection system. Due to the lower heat value it causes lower full load characteristics of the engine.

RME can be aggressive against different materials, especially sealings, paints, rubber and other synthetics. It provokes a quicker oil dilution and deterioration of oil quality – more frequent oil exchange intervals as well as special oils are recommended.

RME, as all bio-fuels, is bio-degradable and CO₂-neutral.

Thanks to the oxygen content (10.7%) RME can lower the emissions of CO, HC including aromatics, aldehydes & ketones, [1].

RME produces a lower part of insoluble fraction (INSOF) in the particle mass and it often causes higher particle number concentrations in the nuclei mode (the lowest invisible size range <40nm) due to the spontaneous condensates, [1, 2].

The soluble fraction (SOF) with RME has the lowest mutagenous effects, [1].

The comparison of PM-emissions with RME and standard fuel depends on the working collective of the engine, since the operating regime influences the SOF/INSOF proportion and RME provokes more deposited soluble organic fraction (SOF).

In an extensive study of the US EPA, [3], with blending of 20% soja oil methyl ester (SME), following average changes were found:

NO_x + 2%; PM -10,1%, HC -21,1%; CO -11.0%

In contrary to that a Swiss research program, [4], found no significant improvements of emissions CO, HC & PM with 15% RME blends. The statements about PM-composition and about NP-count concentrations could be confirmed:

- EC share of the PM: significantly less than for diesel fuel, decreasing with increasing RME-content.
- OC share of the PM: much more than with diesel fuel and increasing with RME content.

- Smoke emission: noticeably diminished black smoke and opacity, both steady-state and transient conditions, improving with higher RME content.
- Particle size distribution: consistent curtailment of particles larger than 80 nm but some increase, at all operating states, of particles smaller than 80 nm.

Further important information about biofuels can be found in [5, 6, 7, 8].

DPF regeneration, soot oxidation & structure

The impact of blending 20% SME (soya methyl ester) to Diesel fuel on the regeneration of a catalyzed DPF was investigated in [9] (US) on a Cummins 6 cyl. 5.9 L engine. The used tools were: the traditional engine diagnostics for injection, combustion and limited emissions, as well as analytics of soot composition and structure. It was shown, that biodiesel blending alters the composition of the particulate matter (PM), as well as the nanostructure of the primary soot particles and thereby alters the regeneration behaviour of DPF. The inclusion of biodiesel in the base fuel reduces the temperature required to initiate the regeneration. This is attributed to the changed nanostructure and oxidation reactivity of the primary soot particles, yielding a more amorphous soot structure, which enhances the soot oxidation.

In [10] the same researchers group focused on the mechanisms of oxidation of biofuel soot (B100, SME) and confirmed that due to different structure and oxygen content the B100 soot is more reactive and undergoes a different oxidation process, than the soot derived from Diesel fuel (ULSD & FT). By means of partial oxidations and a detailed analysis of soot structure it was shown that the crucial mechanism by which biodiesel soot enhances oxidation is a structural transformation of the surface of the soot conglomerates in the early stage of oxidation, whereby the initial surface oxygen groups are more important than the initial structure and pore size distribution.

In [11] the effects of biodiesel blends (B20 & B100, SME) were investigated on a Cummins engine 5.9L with a catalyzed CRT (continuously regenerating trap). Significant lowering of the balance point temperature (Δt 45°C with B20 Δt 112°C with B100), as well as an increase of regeneration rate was found. If it would be possible to renounce on active regeneration system due to the use of biofuels, this would mean a supplementary energy saving.

Also the researchers in [12], investigated B50 & B100 (seed oils methyl ester) on two engines, found differences of morphology (SEM), differences of chemistry and more easy passive regeneration of DPF with biocomponents. It is suggested that potassium contained in the investigated oils might assist in formation of inorganic substances, which would catalytically lower the ignition temperature of the soot.

Objectives

The objective of the present paper is to summarize the most important results obtained by AFHB and by the collaborating laboratories EMPA- and JRC-analytics during the project activities with RME.

There is information about:

- emissions without & with DPF with Bxx,
- influences of RME on PM & PN, EC & OC,
- soot loading, regeneration and anatomy of DPF,
- secondary emissions and traces with SCR & RME,
- DOC light-off with Bxx,
- TEQ with Bxx, SCR & DPF (particle mass from some characteristic test series were investigated at JRC Ispra on PAH, TEF and TEQ).

Additionally several practical remarks and recommendations have been given, which would help the user of biofuels FAME's to attain a satisfactory engine operation with possibly lowest exhaust emissions and energy consumption.

TESTED ENGINES & FUELS

Engines

Liebherr engine for construction machines was used during the projects with RME. Due to the collaboration with the engine manufacturer an access to the engine ECU was given, which enabled the setting of certain parameters and of the full load characteristics.

For testing of SCR-, or (DPF+SCR) - systems an IVECO engine (Euro 3) was used. This engine is attached to a dynamic brake, which makes possible to perform all kinds of dynamic testing.

The engines data are summarized in [Table 1](#).

[Figure 1](#) shows Liebherr engine and [Figure 2](#) the Iveco engine in the laboratory for IC-engines, University of Applied Sciences, Biel-Bienne.

Table 1: Data of the research engines in the RME-research programs

Manufacturer	Liebherr Machines Bulle S.A., Bulle/Fribourg	Iveco, Torino Italy
Type	D934 S	F1C Euro 3
Displacement	6.36 Liters	3.00 Liters
RPM	2000 rpm	max. 4200 rpm
Rated power	111 kW @ 2000 rpm	100 kW @ 3500 rpm
Model	4 cylinder in-line	4 cylinder in-line
Combustion process	direct injection	direct injection
Injection system	Bosch unit pumps	Bosch Common Rail (CR) 1600 bar
Supercharging	Turbocharger with intercooling	Turbocharger with intercooling
Emission control	none (exhaust gas aftertreatment according to the requirements)	none
Development period	2005	until 2000

DPF installed in the exhaust line

Figure 1 - Investigation of DPF on the LIEBHERR engine D934

Figure 2 - (DPF + SCR) system on the Iveco engine F1C

Table 2 - Fuel properties as per EU-standards and further analysis of the test fuels

		Diesel	RME
Density at 15°C	g/m	0.832*	0.845*
Viscosity at 40°C	mm ² /s	2.0 – 4.5	4.6*
Flash point		above 55°C	143°C
Cloud point		max -10°C	-
Filterability CFPP		max -20°C	-15
Ash	%	max 0.010	Traces
Sulfur	ppm	<10	1.3*
Cetane Number		51	56
Calorific value	MJ/kg	42.7	37.2
C fraction	in %	86.7	77.5
H fraction	in %	13.3	11.8
O fraction	in %	0	10.7
Air _{min}	kg/kg	14.52	12.49
Boiling range 10-90% °C		180 - 340	315 - 360

* measured values

Fuels

Following base fuels were used for the research (Table 2):

- Shell Formula Diesel fuel Swiss market summer quality (10 ppm S) according to SN EN 590
- Rapeseed Oil Methyl Ester RME from Flamol, Berne, CH

Table 2 represents the most important data of the fuels according to the standards and the analysis certificates.

It can be remarked, that there are differences of density, heat value, stoichiometric air requirement and boiling range, which have influences on the engine operation and especially on the full load parameters. These changing fuel parameters are taken into account by the evaluation of measurements. The resulting fuel parameters for the blend fuels are represented in Table 3.

For the reasons of market the testing of fuels with low RME blending ratio (B7 & B10) is required. This usually makes no difference of results to the pure Diesel fuel. Therefore for the reasons of research higher blending ratios (B20, B30 & B100) were used in order to may indicate the maximum differences.

Table 3 - Principal parameters of the blend fuels

	$\rho(20^{\circ}\text{C})$ [kg/dm ³]	Hu [MJ / kg]	Air min [kg _A / kg _F]
Diesel	0.832	42.70	14.52
RME 7	0.835	42.30	14.38
RME 20	0.842	41.55	14.10
RME 30	0.846	40.98	13.89
RME 100	0.880	37.20	12.49

TEST METHODS AND INSTRUMENTATION

Engine dynamometers and standard test equipment

Figure 3 represents the special systems installed on the engine, or in its periphery for analysis of the legislated and non-legislated emissions on Iveco engine. The positions of measured control parameters (p, T) in different positions of the air- and gas path are marked.

For research different sampling positions (SP) in the exhaust system were used. For combined exhaust aftertreatment systems (DPF + SCR) a DPF is mounted between SP1 and SP2, [13, 14, 15].

The SCR-system has its own ECU and control sensors (NO_x and temperature).

The test stand of Liebherr engine is equipped with a eddy current dynamometer, with similar measuring positions of the control parameters (p, T) and uses the same measuring systems for standard and for non-legislated components.

Figure 3 - Engine dynamometer and test equipment for testing of SCR-systems on Iveco engine (engine variant with EGR)

Test equipment for exhaust gas emissions

Measurements are performed according to the Swiss exhaust gas emissions regulation for heavy duty vehicles (Directive 2005 / 55 / ECE & ISO 8178):

- Volatile components:

- Horiba exhaust gas measurement devices
Type VIA-510 for CO_2 , CO , HC_{IR} , O_2 ,
Type: CLA-510 for NO , NO_x
- Amluk exhaust gas measurement device Type FID 2010 for HC_{FID} ,
- NH_3 & N_2O :
With SCR several unlimited components can be produced. The slip of gaseous components: ammonia NH_3 and nitrous oxide N_2O was measured by means of:
 - Siemens LDS 6 Laser Analyzer 7MB 6021, NH_3
 - Siemens ULTRAMAT 6E 7MB2121, N_2O
 - Eco physics CLD 822 CM hr with hot line for NO , NO_2 , NO_3 , NH_3 .

- FTIR (Fourier Transform Infrared) Spectrometer (AVL SESAM) with possibility of simultaneous, time-resolved measurement of approx. 30 emission components – among others: NO , NO_2 , NO_x , NH_3 , N_2O , HCN , HNCO .

Particle counts & size analysis

To estimate the filtration efficiency of the DPF, as well as to detect the possible production of secondary nanoparticles, the particle size and counts distributions were analysed with following apparatus:

- SMPS – Scanning Mobility Particle Sizer, TSI (DMA TSI 3071, CPC TSI 3025 A)
- Sample preparation:
 - MD19 tunable minidiluter (Matter Eng. MD19-2E) – heated up to 80°C
 - Thermoconditioner (TC) (i.e. MD19 + postdilution sample heating up to 300°C).

The nanoparticulate measurements were performed at the constant speed (warm) with SMPS. During the dynamic engine operation the CPC was used.

TEST PROCEDURES ON ENGINE DYNOS

On both engine dynamometers stationary operating points (OP), so called steps-tests were performed. An example is given in Figure 4.

In VERT Filter Test (VFT) generally 4-points tests are used, [16, 17].

The VERT secondary emissions tests VSET consist of repetitions of ISO 8-points tests.

In other tests certain OP's were chosen or added according to the research objectives. Particularly to mention is the addition of 3 operating points between OP3 and OP4 to better recognize the urea switch on/off of the SCR-system, [14, 15].

All steps-tests were performed with a warm engine and for each research task always in the same sequence and with the same operating duration of the OP's.

After analyzing the backpressure of the (DPF+SCR) system at stationary operation in the Iveco engine operation map it was decided to limit the operation range, [Figure 4](#). In this limited engine map (LEM) different steps-tests were defined.

With the biofuels, or bio-blend-fuels the calorific value of the fuel changes and so does the full load torque (FL). In the test programs the part load OP's were generally driven with the same power (speed & torque) and for the FL OP's only the required speed was kept (torque resulted from the fuel). The injection characteristics of the engines were not changed, which represents the usual situation in the market application.

Figure 4 - Limited engine map of the IVECO F1C engine and 8 points test according to ISO 8178

EXHAUST AFTERTREATMENT

Presented results are obtained with following exhaust aftertreatment devices:

- Diesel oxidation catalyst (DOC, Pt) with catalyzed soot filter (CSF, SiC, $\text{TiO}_2 + \text{V}_2\text{O}_5$) – designated as (DOC+CSF), or GFM
- CSF alone
- uncoated DPF (for active regenerations, SiC) also designated as SFM; (the short names GFM & SFM are especially useful for the section “DPF anatomy”)
- Vanadium based SCR (VSCR)
- combined system: cDPF (Cordierite, Pt) upstream + VSCR downstream.

The used exhaust aftertreatment devices were retrofit parts and they were adapted by the supplier to the size of the engine. In certain cases for frequent research of soot loading and regenerations smaller DPF-units were used and the operating range of the engine was respectively adapted in view of the admissible backpressure.

RESULTS

Emissions with & without DPF

From different test series performed with biofuels with and without DPF a typical example with catalytic DPF (CSF) and with several blending ratios (B0, B7, B20, B30 and B100) was chosen.

The DPF-system (DOC+CSF) was investigated in the VERT 4 pts-test and was used for several tests of regenerations with biofuels.

The comparisons of emissions with different fuel blends and with / without this DPF-system, [Figure 5](#), show:

Without DPF lowering of CO, HC and lowering of PM at high-load OP's with increasing bio-content. The NO_x -values increase with Bxx, which is a fact generally confirmed by the own and foreign research.

With DPF there are: elimination of CO, strong reduction of HC and an increase of the $\text{NO}_2 / \text{NO}_x$ -ratio. With increasing bio-content this $\text{NO}_2 / \text{NO}_x$ -ratio decreases at part-load OP's. There is a clear catalytic influence of the tested DPF (DOC + CSF).

The investigated DPF shows a very good reduction of particle mass PM.

[Fig. 6](#) shows the comparisons of SMPS PSD-spectra with all fuel blends (except of B7 for graphical clarity) at the high load OP (1100rpm/480 Nm).

There is a tendency (w/o DPF) especially with B100, to emit high PC in nuclei mode (size spectrum below 30 nm) and lower PC-concentrations in accumulation mode (30-300 nm). With DPF there are also higher PC-concentrations and higher values of penetration with B100 – more spontaneous condensates.

These tendencies are similar for other OP's and there is also a certain logical sequence of curves with increasing share of biocomponents.

The presented DPF system (DOC+CSF) was chosen on purpose smaller, than necessary for the research engine to enable a quicker soot loading. Thanks to that it was possible to approach the limit of maximum spacial velocity of this filter, when the NP-blow-through starts to take place. The operating points of the 4 pts. test were adequately set at lower engine speeds 900 rpm and 1100 rpm.

Figure 5 - Comparison of emissions with different fuel blends and with/without DPF-system (DOC+CSF) engine: Liebherr D 934 S

Figure 6 - SMPS PSD - spectra & penetration values with different fuel blends at (1100rpm/480Nm), with/without DPF coated with DOC, engine: Liebherr D934S

Table 4 - Comparison of trapping efficiencies of counts 20-300 nm (PCFE) with different fuel blends, with / without (DOC+CSF), engine: Liebherr D 934 S; fuels: B0, B7, B20, B30 & B100

		PCFE [%]			
Operating points					
speed	rpm	900	900	1100	1100
torque	Nm	473	315	320	480
ulsd		98.63	99.99	100.00	98.55
RME7		98.09	99.92	99.94	97.83
RME 20		98.95	99.97	99.98	97.98
RME 30		97.40	99.97	99.98	97.62
RME 100		95.45	99.75	99.78	97.06

Table 4 summarizes the particle count filtration efficiencies (PCFE) for low and high load OP's. The tendency of a lower filtration efficiency with higher gas flow (higher OP), as well as the tendency of lower filtration efficiency with B100 (more spontaneous post condensation) are confirmed. The filtration material of the investigated DPF fulfills the highest quality requirements of VERT and the requirements of Swiss LRV (OAPC).

Operation above the limit of spacial velocity provokes the NP-break-through and deteriorates the filtration efficiency PCFE.

Coulometry EC & OC

Bio components have an impact on the composition of the particle matter (PM) emitted by the engine.

At the operating points: 1500 rpm / 10% and 1500 rpm / 80% single samples of particulate matter for coulometry were taken for Diesel and RME (without DPF) on special quartz measuring filters. The coulometric analysis of filter residuum was performed by the SUVA analytical laboratory.

The standard procedure consists of thermal extraction of SOF with N₂ at 500°C during 8 min and after that of the EC-oxidation with O₂ at 650°C.

Since RME is a fuel, which produces higher portion and heavier SOF, it was decided to increase the temperature of the first thermal N₂-extraction to 800°C for all samples.

The coulometric results EC, OC & TC – all values responding to 3 min sampling time on the entire measuring filter and to the same sampling conditions – are represented in [Fig. 7](#).

It can be remarked, that:

- higher engine load produces less OC and more EC, i.e. “drier” soot
- RME causes higher portion of OC (lower portion of EC), which is particularly pronounced at lower engine load
- RME reduces the total PM at higher engine load, especially due to the reduction of EC.

Figure 7 - Influences of engine load on EC/TC-ratio for different fuels

Passive Regenerations

The results of soot loading procedures give important information about the effects of biocomponents.

The soot loading of the DPF's was performed up to $\Delta p \approx 100$ by means of a rectangular cycle 0-500 Nm & 800-1100 rpm, which was the best compromise to produce PM without, or with minimum passive regeneration.

All loading procedures, except of B100, were performed in identical way. For B100, which yielded very slow soot loading, periodical switch-off's of the engine were applied to cool-down the exhaust system and to minimize the oxidation.

The soot loading with increased bio-content needed always a longer time period. This is represented for (DOC + CSF) and for CSF in [Figure 8](#).

The final particle mass, as product of engine-out emission and time, increases. Nevertheless the oxidized portion of PM during the soot loading procedure and the PM remaining in the DPF at the end of the soot loading procedure are unknown.

The reasons for slower soot charging with biocomponents are:

- lower engine-out PM-emissions,
- higher reactivity of PM and partial oxidation during soot loading with DOC,
- with high bio-content (here B100) reaching of exhaust gas temperature, which is nearer to the temperature-window of the highest NO₂-production in DOC, easier NO-NO₂ oxidation with B100 and the intensified NO₂-continuous regeneration.

Figure 8 - Backpressure during soot loading cycle

After the soot loading each regeneration attempt was performed in the same way: stepwise increasing of engine torque at a constant speed.

In Figure 9 – the comparison of regenerations 7, 11 & 10 (B0, B20 & B100 with DOC + CSF) shows with B100 following tendencies, which would not be visible with B20:

- there is more NO₂ with B100 with a peculiar NO₂ generation behaviour in the subsequent load steps,
- the intensity of regeneration with B100 from 5th step (approx. 300°C) is higher; the drop of backpressure quicker than with the other fuels,
- with B100 the N₂O peak in 2nd step is a little bit higher, than with B20 (7ppm / 5ppm) and after start of regeneration NH₃ is produced with B100 up to 6ppm (during 1/2h), while with B20 it stays at zero-level; all this confirms the increased ractivity of B100.

The specific NO₂ production with B100 was confirmed with all used measuring systems and the authors explain it with the combination of different effects:

- the exhaust temperature with B100 is lower i.e. nearer to the range of the maximum of NO₂-production; the authors believe, that due to the general higher reactivity of B100-exhaust components, the temperature window of NO₂-production is larger,
- there is more NO_x with B100 (up to 20% - not plottet here),
- there is more easy NO-NO2 oxidation,
- in the steps 5 & 6 there is the most pronounced backpressure drop with B100, which means that most of the soot was burned away,
- in the further steps 7, 8 & 9 periodical effects are observed:
 - a) shortly after load increase NO₂ decreases because of higher spatial velocity and shortly increased availability of more PM and NO₂- consumption for oxidation. In this period the NO and NH₃ increase,
 - b) in further part of the load step the DPF attains the new thermal equilibrium the NO₂ starts to increase and NH₃ decreases. NO₂ and NH₃ are anti-correlated,
 - c) the slight generation of NH₃ is possible from NO & H₂O due to the increased reactivity of B100 – exhaust gases.

To deepen, or to confirm the demonstrated reaction mechanisms more research would be necessary. Nevertheless the authors are sure to have demonstrated an important and specific feature of emissions with B100.

Table 5 summarizes the domains of temperatures when the start of backpressure drop is visible in the investigated regeneration variants. There are higher temperatures necessary with less catalytic activity and they decrease with growing bio-content in fuel.

Figure 9 - Regenerations with (DOC + CSF) & B0, B20 & B100

Table 5 - Domains of temperatures with the beginning backpressure drop of the investigated regeneration cases

DOC + CSF		
B0	B20	B100
356-368°C	356-363°C	342°C
at step 5	at step 5	at step 5

CSF	
B0	B20
454-464°C	447-455°C
at step 7	at step 7

DPF anatomy

Some DPF substrates, which were operated only with one sort of fuel (each one) were cut at EMPA Laboratories and analysed for composition and distribution of soot and ashes.

Each substrate, except of B100, experienced 3 soot loadings (up to $\Delta p = 100$ mbar) and regenerations before being cut for the analysis. With B100 the substrates were charged only once. The uncoated substrate (SFM) accumulated so much combustible PM with high heat value, that the DPF was overheated and damaged during the regeneration attempts. This substrate was cut after this trial and was not used for any other tests.

Macroscopic and scanning electron microscope (SEM), as well as transmission electron microscope (TEM) and X-Ray diffraction studies were carried out to investigate the nature and properties of particulate matter (PM) deposited in six diesel particulate filters (DPFs) made of SiC and operating with the same HD engine (Liebherr D 934 S) as follows: with 100 % rapeseed methyl ester (RME) biofuel (B100), diesel fuel (B0) and a blend of 20% RME and 80% diesel (B20). Three out of the six DPFs (named hereafter GFM) were catalytically coated with $\text{TiO}_2/\text{V}_2\text{O}_5$, the other three (named hereafter SFM) were uncoated. The investigated PM deposits were either extracted from sectioned DPF channels or studied 'in situ', as deposited.

Figure 10 - Scanning electron microscope SEM representative images of inlet and outlet (OUT) channels of the GFM- and SFM-DPFs showing a soot cake deposited on the inlet channels of the GFM-B0-DPF, the GFM-B20- and SFM-B20-DPFs. The B100-DPFs show no soot cake deposition on the channel walls.

The B100-DPFs show very little soot deposition, opposite to B20 and B0 which exhibit abundant soot deposits. This observation applies for both GFM and SFM types of DPFs. Soot in all three GFM-DPFs, as well as in SFM-B20 and SFM-B0 forms a cake deposited on the inlet channel walls (Fig. 10). In GFM-B100, the soot cake is thinnest or non-existing. The soot cake deposited on the GFM-B100-DPF cannot hold, at some stage, on the walls of the channels,

probably because of the nature and amount of the underlying material composed of ash and other particle components. The latter are related to the catalytic coating material and/or to fibrous material mechanically transported from the inlet gasket and the intumescent mat enveloping the DPF-DOC assembly.

Based on TEM imaging, the average size of the individual soot particles in GFM-B100 is 21 nm (mean value of 101 measurements) with minimum and maximum values of 11 nm and 38 nm, comparable and rather on the low side in relation to pure diesel soot. Preliminary results of high resolution (HRTEM) images depicting the internal nanostructure of the primary particles of GFM-B100 soot indicate a relatively higher oxidative reactivity compared to diesel soot, a feature described also in previous research studies.

It is recalled that, based on a series of TEM studies (e.g. [9, 10]), the more amorphous the internal nanostructures of primary soot particles the more easily they can be oxidized. Moreover, smaller particles show higher response to oxidation (due to higher surface to volume ratio) than larger ones.

More amorphous structure (higher oxidative reactivity)

Figure 11 - HRTEM images of a representative soot particle from the GFM-B100-DPF. In (B) the image is processed in 'Photoshop' to render the fringes arrangement more discernible. The dashed line marks the particle core with a more amorphous structure.

Locally, a higher degree of crystalline order is observed by regular repetition of graphenes (up to 10 alternating layers in 1-2 nm wide packages) in a crystalline (graphite-like) structure (white rectangle in Figure 11A). Based on macroscopic, as well as optical microscopic and SEM observations on a series of samples from the dissected DPFs, the B100-DPFs contain significantly more ash compared to B20 and B0 implying that besides lubricating oil, RME-biofuel is the major contributor of ash production. This observation is more pronounced with the GFM-B100-DPF type, probably because the extremely high temperatures experienced by the SFM-B100-DPF obscured the original PM deposition material in this filter. In the B0- and B20-DPFs of both GFM and SFM types, ash occurs onto the channel walls underneath the soot cake. Ash is found on the channel walls of GFM-B100-DPF implying that it occurred also underneath the thinner soot

cake, at the time when this was deposited on the channel walls.

Newly formed V-O-bearing long prismatic nanocrystals occur in abundance within fissures of the catalytic coating layer, as well as onto the coating layer surface in both inlet and outlet channels of all three GFM-DPFs. Their size and shape is such that they can escape to the ambient air.

Al-Si-O-, as well as Ca-Al-Si-O-fibres deriving from the gasket on the outer inlet surface of the DPF and the intumescent mat, respectively, occur inside all six DPFs, often mechanically mixed with ash. In the overheated SFM-B100, the Ca-Al-Si-O-fibres are locally molten forming droplet-like to spherical formations.

Based on SEM imaging and qualitative energy dispersive X-ray analyses (EDX), the material underlying the soot cake in the three GFM-DPFs, which are catalytically coated, consists of ash (Ca, S, P, O, Mg, K), catalytic coating material and its reaction V-O-long prismatic crystal products, as well as gasket (Al, Si, O) and intumescent mat (Ca, Al, Si, O) fibres. In the SFM-DPFs, the deposition material other than soot consists of ash (Ca, P, S, Zn, Si, Mg, K, Al, V, Y, O), gasket (Al, Si, O), as well as intumescent mat (Ca, Al, Si, O) fibres.

Needle-like to worm-like formations consisting mainly of Si and occasionally variable amounts of Al, O, C occur attached to the filter wall material of all three SFM-DPFs. Their formation may be related rather to the SiC wall material. In the SFM-B100-DPF, which has locally seen very high temperatures (>1200 °C), both filter material, as well as deposited ash PM and intumescent mat melted, at least partly. The filter material gave rise to new fibrous, platy and spherical particles of SiC composition.

Secondary emissions and traces (whole exhaust at tailpipe)

Secondary emissions and traces are investigated as a part of VERT / LRV quality verification procedure for DPF- and SCR-systems. The sampling and analytics are performed by a specialized team of EMPA Analytical Laboratory for Organic Chemistry. There are 3 groups of secondary emissions, which are known as very carcinogen and toxic:

- Polycyclic Aromatic Hydrocarbons PAH, 12 substances ^{*)}, [µg/L],
- Nitro-PAH, 22 substances ^{*)}, [µg/L],
- Polychlorinated Dibenzodioxins PCDD and Polychlorinated Dibenzofurans PCDF, 33 substances ^{*)}, [pg/L].

A VERT Secondary Emissions Test was performed with biofuels B100 and B30 and with urea based SCR as well as with combinations of SCR and biofuels (without DPF).

Samples were taken continuously proportional to the actual gas flow (aliquot) from the undiluted hot exhaust gas. A volume of typically 5-8 Nm³ was collected through a validated sampling arrangement over 200 Minutes driving cycle time (2 full ISO 8178/4 C1-cycles).

The sampling device consists of a sampling sonde, cooler, condensate separator, filter stage and two-stage adsorber unit (XAD-2). The sampling apparatus, made from glass is extensively cleaned and heated to high temperatures prior to each sampling (heating in steps up to 450°C). Using marked PCDD-standards in this investigation the recovery was found to be 65 ± 10 %.

The same sample was used to determine the PCDD/F as well as the PAH.

PCDD/F-analysis was performed by separating the toxicologically relevant PCDD/F isomers using gas chromatography followed by high resolution mass spectrometry.

After chromatographic work-up quantitative analysis of the PAH and Nitro-PAH were also performed using capillary gas chromatography high resolution mass spectrometry.

PCDD/F-Analysis

The following graph, [Figure 12](#), shows the overall results.

There seems to be a slight indication of increase when biofuels are used without SCR. This is more pronounced with some individual compounds like the Seveso Dioxin. When SCR is used these toxic substances are clearly reduced with both fuels.

Figure 12 - Emission factors for the weighed sum of 2, 3, 7, 8-substituted PCDD/F (TEQ-sum) in pg per liter fuel, Iveco FIC E3.

^{*)}analyzed in presented tests

PAH-Analysis:

PAH were analysed from the same sample as the PCDD/F. The following graph, Fig. 13, summarizing the data for the carcinogenic PAH according to the IARC-definition shows the overall results.

Figure 13 - Emission factors for the sum of carcinogenic PAH (IARC) in µg per liter fuel, Iveco FIC E3.

It can be summarized, that:

- Biofuels B100 and B30 had little influence on NO_x, NO₂/NO_x, CO, CO₂ and consumption of fuel, however they reduced HC by about 50 %, PAH by 52-78% % and Nitro-PAH by 50-70 %
- The Urea based SCR reduced NO_x on average by about 85 % and eliminated NO₂
- SCR also reduced HC, PAH and Nitro-PAH very effectively but had no influence on CO
- The side effects of the SCR technology can be emissions of NH₃, N₂O and HNCO. These were visible but not very pronounced
- Formation of dioxins and furanes was visible but not promoted by these technologies
- The combination of SCR and biofuels had no negative side effects – on the opposite a further reduction of PAH and PCDD/F could be observed.

DOC light-off with biocomponents

In order to investigate the DOC light-off load steps were performed at different engine speeds (900, 1100 & 1300 rpm) with B0, B20 & B100. These load steps with 5 min step duration were conducted up and down to see the possible effects of hysteresis.

Following findings can be pointed out:

With increased bio-content there is a higher NO_x- and lower HC-emission. During the load jump from the 1st to the 2nd step, there are:

- elimination of CO, equal for all fuels,
- lowering of HC with B100, while increasing with B0 in the 2 minutes after load jump – most probably due to the starting oxidation of HC with B100, (CO & HC not demonstrated here),
- quicker increase of NO₂ with B100 which indicates an easier light-off with B100.

Similar tendencies are also visible with B100 at higher engine speeds.

The differences are less pronounced due to higher flow velocities through the catalyst.

The representation in function of exhaust temperature, Fig. 14, confirms that there is light-off for NO₂ starting with B0 at approx. 220°C, with B20 at 200°C and with B100 at 170°C.

For HC the lowering in the 1st step with B100 is confirmed, which indicates the light-off for HC at approx. 170°C. (As light-off in this context the visible start of reaction is understood).

Figure 14 - Influences of fuels B0, B20 & B100 on DOC light-off for NO₂ in steps tests; Liebherr D 934S A6

The consideration of NO₂- and HC-plots in function of exhaust gas temperature at increasing engine speed, allows to remark, that there is a little influence of increasing gas flow and increasing spacial velocity (SV) on the light-off. With higher engine speed the start of increasing NO₂, or decreasing HC is moved to higher exhaust gas temperature (in the range of $\Delta t \approx 10^\circ\text{C}$).

With the chosen duration of steps (5 min) there were differences of results between the tests with increasing or decreasing load.

It can be generally remarked, that during the down-steps there is higher temperature level, which causes the more intense catalytic conversion of CO, HC & NO₂ in the lower steps. These temperature differences caused by different ways of conducting the tests are due to the thermal inertia of the system.

The resulting statement is that the results in all steps tests (like ISO 8178, or ESC) are connected to the time-schedule of the test procedure.

PAH & TEQ of particle mass with biofuels

Particle matter (PM) emitted by the engine at different operating points (OP), with different fuels (B0, B30 & B100) and with different exhaust gas aftertreatment was sampled and analyzed at the JRC laboratories.

14 Polycyclic Aromatic Hydrocarbons (PAH) were determined in the filter samples by means of the High Pressure Liquid Chromatography (HPLC) and the toxicity equivalence (TEQ) according to the EPA method was estimated.

The analyzed PAH and their toxicity equivalence factors are represented in the following [Table 6](#):

Table 6 - Toxicity equivalence factors of different PAH's (according to US EPA 1994)

Compound	TEF
Fluorene	0,001
Phenanthrene	0,001
Anthracene	0,001
Fluoranthene	0,010
Pyrene	0,001
Benzo[a]anthracene	0,100
Chrysene	0,010
Benzo[b]fluoranthene	0,100
Benzo[k]fluoranthene	0,100
Benzo[e]pyrene	0,002
Benzo[a]pyrene	1,000
Indeno[1,2,3-CD]pyrene	0,100
Dibenz[a,h]anthracene	1,000
Benzo[ghi]perylene	0,010
Coronene	0,010

Particle matter was sampled on the Iveco F1C engine at a part load (PL) and full load (FL) operating points with different exhaust aftertreatment systems according to the following matrix:

	B0	B30	B100
w/o aftertreatment	PL / FL	PL / FL	PL / FL
DPF + SCR	PL / FL	PL / FL	PL / FL
SCR only	PL / FL	PL / FL	PL / FL

PL (part load): 2200 rpm / 32 Nm OP4, 7.4 kW
FL (full load): 2200 rpm / 300-330 Nm OP1, 74 kW

The comparison of TEQ in [ng / mg PM] for all investigated variants of fuel, of aftertreatment and operating points is represented in [Fig. 15](#).

It can be remarked:

- there is generally a lower TEQ at higher OP1,
- with increased bio-content there is a tendency of lowering TEQ at lower OP4 and of increasing TEQ at higher OP1; (some exceptions – especially SCR B0 & SCR B30 at OP4 – are attributed to the measuring and emitting dispersion),
- the SCR alone produces at low load generally higher TEQ as (DPF+SCR),
- at high load with higher bio-content the TEQ of (DPF + SCR) is higher, than of SCR alone – this can be attributed to the condensation effects after DPF, which are promoted by the presence of bio-components and a seeding effect of the secondary NP from SCR.

The view on TEQ-comparisons in [$\mu\text{g} / \text{kWh}$], (not represented here), allows further statements:

- SCR has a tendency to slightly lower the TEQ-values, comparing with no aftertreatment,
- DPF is the best method to eliminate PAH and to minimize TEQ.

Figure 15 - Toxicity Equivalence TEQ [ng / mgPM] with biocomponents (RME), SCR and DPF, Iveco F1C E3

CONCLUSIONS

The most important conclusions, according to the investigated topics and the performed working packages, can be listed as follows:

Emissions with & without DPF

- RME lowers CO and HC and increases NO_x at all operating points, it lowers PM at higher engine loads and increases PM at lower engine loads. RME has no effect on specific energy consumption.

- RME moves the PSD spectra to smaller sizes and increases the nuclei mode due to spontaneous condensates,
- with RME there is a higher portion of condensates (SOF) at low load and idling,
- RME causes higher portion of OC (lower portion of EC), which is particularly pronounced at lower engine load,
- RME reduces the total PM at higher engine load, especially due to the reduction of EC,
- with (DOC + CSF) there is a clear catalytic influence: elimination of CO, strong reduction of HC and increase of the $\text{NO}_2 / \text{NO}_x$ -ratio,
- the tested DPF reaches the nanoparticle counts filtration efficiency PCFE up to 99.99%,
- using of pure biofuel B100 also lowers the PCFE due to post condensation effects and increased penetration of smallest particles, except of that the lower blends (B7, B20 & B30) have no impact on PCFE.

Regenerations with (DOC+CSF)

- with the same soot loading procedure the necessary time to load the DPF for $\Delta p = \text{idem}$ increases with the bio-content of fuel (here for $\Delta p \approx 100 \text{ mbar}$: B0 1,7h; B20 2,5h; B100 27h),
- the reasons for slower soot charging with biocomponents are:
 - lower engine-out PM-emissions,
 - higher reactivity of PM and partial oxidation during soot loading with DOC,
 - with high bio-content (here B100) lowering of exhaust gas temperature to the temperature-window of the highest NO_2 -production in DOC, easier NO - NO_2 oxidation with B100 and the intensified NO_2 -continuous regeneration.

DPF anatomy

- with RME blend fuels more amorphous (reactive) structures of soot are remarkable,
- with higher RME portion there is less soot deposited in the filter, but there are more elements creating ashes, like: Zn, Mg, Ca, K, Si, P, S.

Secondary emissions traces with SCR & RME

- Biofuels B100 and B30 reduced HC by about 50 %, PAH by 52-78% % and Nitro-PAH by 50-70 %
- SCR reduced HC, PAH and Nitro-PAH very effectively but had no influence on CO
- The combination of SCR and biofuels had no negative side effects – on the opposite a further reduction of PAH and PCDD/F could be observed.

DOC light-off with biocomponents

- due to the higher reactivity of RME the fuels with higher bio-content cause an easier light-off of the DOC,
- with B100 the NO - NO_2 -oxidation and the HC-conversion start already at approx. 170°C ,
- with higher engine speed (higher spacial velocity) the start of increasing NO_2 , or decreasing HC is moved to higher exhaust gas temperature (here $\Delta t \approx 10^\circ\text{C}$),
- due to the thermal inertia of the engine-exhaust-system there are differences of emission results by conducting the steps test with increasing or with decreasing engine load,
- the emissions results in all steps tests are connected to the time-schedule of the test procedure.

PAH & TEQ of the PM with biofuels

- DPF has a positive effect of reducing PAH & TEQ of the PM,
- at low-load operating conditions without DPF there is a tendency of higher TEQ (due to presence of B(a)P),
- higher biofuel content has with DPF no influence on TEQ,
- without DPF there is a tendency of lowering TEQ at low load and increasing TEQ at high load,
- no differences concerning TEQ could be remarked for catalytic and non-catalytic DPF's; this result nevertheless must be limited to the present engine and the present operating conditions,
- SCR alone has no influence on TEQ.

REMARKS & RECOMMENDATIONS

The present investigations did not concern the long term effects:

- possible coinfluences of bio-fuels with lubricating oil,
- possible deactivation of catalytic activity in case of too high content of sulphur, phosphorus and dirt if the bio-fuel quality does not correspond to the actual norms,
- possible influences on deposits and material degradation in fuel system, in combustion chambers and in the exhaust aftertreatment of the engine.

The fulfilment of biofuel quality requirements depends on supplier, transport, stocks conditions and time of storage.

Biocomponents have limited durability (validity) time because of oxidative degradation, water absorption and possible bacteria befall [18, 19, 20]. For these reasons the user of biofuel is recommend to periodically control the quality of supply chain and of the fuel.

A recently published study of a US industrial consortium, [21], about the impact of biodiesel impurities on the long-term

behaviour of exhaust aftertreatment elements showed, that due to diffusion of Alkali Metals (K, Na, Ca) into the ceramic materials of DOC, DPF and SCR, the material resistance of these elements is reduced significantly during the long duration operation.

Following recommendations can be given for the use of biofuels with DPF systems:

- the filtration efficiency is not influenced by the biofuel, but there can be artefacts of spontaneous condensates after DPF,
- performing filtration quality tests, like is recommended with a standard fuel,
- for long life of catalytic coatings and for sustainable engine operation control periodically the quality of fuel and fuel supply chain,
- consider the general recommendation for the engine: durability of injection system and of lube oil; power reduction with fuels with lower heat value; limits of electronic regulation with changed heat value of fuel.

The recommendations for the users of SCR systems with biofuels concern the fuel quality and the sustainability of the engine operation and are the same, as mentioned above for DPF.

Generally plan more frequent oil change and inspection/maintenance intervals.

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ABBREVIATIONS

AFHB	Abgasprüfstelle FH Biel, CH
BAFU	Bundesamt für Umwelt, CH (Swiss EPA, FOEN)
BfE	Bundesamt für Energie
BioExDi	<u>B</u> io <u>f</u> uels <u>E</u> xhaust Systems <u>D</u> iesel
Bxx	bio-blend fuels with xx vol. % of RME
CFPP	cold filter plugging point
CLD	chemoluminescence detector
CPC	condensation particle counter
CR	common rail
CSF	Catalytic Soot Filter
DC	Diffusion Charging Sensor
dePN	de Particles + deNO _x
DI	Direct Injection
DiSC	Diffusion Charge Size Classifier
DMA	differential mobility analyzer
DOC	Diesel Oxidation Catalyst
DPF	Diesel Particle Filter
EC	Elemental Carbon (VDI 2465) / European Community
ECU	electronic control unit
EMPA	Eidgenössische Material Prüf- und Forschungsanstalt
EPA	Environmental Protection Agency

ETC	European Transient Cycle
EV	Erdöl Vereinigung
FAME	Fatty Acid Methyl Ester
FE	filtration efficiency
FI	fuel injection (fuel aerosol generator)
FID	flame ionization detector
FL	full load
FOEN	Federal Office of Environment
FTIR	Fourier Transform Infrared Spectrometer
GFM	filter material SiC coated, CSF
HD	heavy duty
IARC	International Association of Research on Cancer
ICE	internal combustion engines
INSOF	insoluble fraction
IR	increase rate, infrared
JRC	EC Joint Research Center
K _x	conversion efficiency of the component "x"
LD	low duty
LDS	Laser Diode Spectrometer (for NH ₃)
LEM	limited engine map
MD19	heated minidiluter NanoMetnanoparticle summary surface analyser (PAS + DC + MD19)
NP	nanoparticles < 999 nm (SMPS range)
OC	Organic Carbon (VDI 2465)
OEM	original equipment manufacturer
OP	operating point
PAH	Polycyclic Aromatic Hydrocarbons
PC	particle counts
PCDD/F	Polychlorinated Dibenzodioxins/Furans
PCFE	particle counts filtration efficiency
PM	particulate matter, particle mass
PMFE	particle mass filtration efficiency
PN	particle number
PSD	particle size distribution
RE	reduction efficiency
RME	rapeseed methyl ester
SCR	selective catalytic reduction
SEM	scanning electron microscope
SFM	filter material SiC uncoated
SiC	silicium carbid
SMPS	Scanning Mobility Particle Sizer
SOF	soluble organic fraction
SP	sampling position
TC	thermoconditioner. Total Carbon
TEF	toxicity equivalence factor
TEM	transmission electron microscope
TEQ	toxicity equivalence
TTM	Technik Thermische Maschinen
UDS	urea dosing system
ULSD	ultra low sulfur Diesel
VERT	<u>V</u> erminderung der <u>E</u> missionen von <u>R</u> ealmaschinen in <u>T</u> unelbau <u>V</u> erification of <u>E</u> missions <u>R</u> eduction <u>T</u> echnologies
VSCR	Vandium based SCR