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Abstract

Due to increasing concern about health effects of fine and ultra-fine particles (nanoparticles) from combustion engines, the diesel particle filter technology (DPF) ^{*)} was extensively introduced to heavy duty and passenger cars in the last years. In this respect, a very important parameter is the irreversible plugging of the DPF with non-combustible ashes. The quality of lubrication oil, especially the ash content has a certain influence on regeneration intervals of diesel particle filters.

In the present study, the effects of different lubrication oils on particle mass and nano-particle size distribution were investigated. The test engine was a modern diesel engine without particle filter system.

A main goal was to find out, how different lubrication oils influence the particulate emissions and the contribution of oil to total particle emissions.

Moreover, first results of a tracing study will be discussed. The comparison of a non-doped lubrication oil with a doped oil should enlighten the contribution of the oil to the particle formation.

It will be shown that beside sulphur content the particle emission is also effected by the composition (e.g. additive packages) of the oils.

^{*)} Abbreviations & references see at the end of paper

Introduction

Quality and consumption of lubrication oil was recognized as an important factor for diesel particle filter technology (DPF) in the Swiss activities about retrofitting of construction machines and city busses with DPF systems ^{[1][2][3][4]*}. Low ash and low sulphur contents diminish irreversible deposits and increase regeneration intervals of DPF's. However, a main disadvantage is a potential negative effect on oil durability. Therefore, oil exchanges are more often necessary.

Lubrication oil has a significant influence. Thus, the effects of different lubrication oils on particle mass and particle size distribution is of major interest. In previous studies of AFHB concerning VERT verification procedure of DPF's ^{[2][3][6][7][8][9]}, on a Liebherr-engine for construction machines, it was already observed that different lubrication oils provoke different particle emissions. It is known that oil lubricating cylinder walls is a particle source in combustion. Another effect is the blow-by, which is recirculated to the intake air of the engine.

Also possible oil losses along the intake valve shafts and in the turbocharger can increase oil consumption.

Therefore further investigation on oil related particle generation are necessary to investigate how different lubrication oils influence the particulate emissions and the amount of oil contribution to total particle emissions.

Experimental

Tests were performed with three commercial oils from Lubrizol (GB) within the bus retrofitting projects of city of Biel and Canton Bern which were supported by the Swiss Federal Office of Environment Forests and Landscape (FOEFL).^[5] Additionally the effect of three further oils was investigated.

For the tracer study the tests were repeated with two doped lubrication oils containing a mixture of the elements Pb, Cd, Ba and Sb. The oil-based solution was added in a 1:58 ratio to the lubrication oil.

In this study a modern VW TDI 1.9 L engine was used for the nanoparticle investigations. Fig. 1 shows the engine used in the measuring laboratory with the sampling points and the partial flow dilution tunnel.

Test-Engine

Engine

Manufacturer:	VW
Type:	AJM
Displacement:	1 896 cm ³
Rated RPM:	4 000 rpm
Rated power:	85 kW
Model:	4 cylinder R
Compression ratio:	18 : 1
Oil capacity:	4.5 dm ³
Combustion process:	direct injection
Injection pump:	unit injectors
Supercharging:	Turbocharger with vario turbine
Emission control:	oxidation catalyst
Model year:	1999

Fuel

Greenenergy ultra low sulphur (ULSD) Diesel fuel (S < 10 ppm) was used for all tests.

The following standard parameters according to SN EN 590,^[10] were used for all tests:

Density (at 15°C):	0.820 - 0.835 g/ml
Viscosity (at 20°C):	2.0 - 4.5 mm ² /s
Flame point:	min 60°C
Cloud point:	max - 32°C
Filtering limit:	CFPP max. - 32°C
Coke residue	:
(Conradson at 10% test residue):	max. 0.05 g/100 g
Ash:	0,003 g/100 g
Sulphur:	max. 0.0010 g/100 g
Cetane index (determined):	min. 52-54

Brake Engine VW TDI Dilution
Schenck tunnel

Fig. 1: VW TDI engine at the test stand

Measuring Equipment

The schematic of measuring set-up is shown in Figure 2.

The following test equipment was used for the measurements of exhaust gas emission:

Volatile components:

- Horiba exhaust gas measurement devices
Type PIR-2000 for CO₂, CO, HC_{IR}
- Beckmann Analyzer CLD Model 955 for NO, NO_x
- Ratfish FID RS 5 for HC_{FID}

Fig. 2: VW TDI engine measuring set-up

Measurement of particulate mass emissions:

- Sampling and dilution:
partial flow dilution tunnel
Matter-Eng. / METAS
- Gravimetry:
Filter temperature: $\leq 52^\circ\text{C}$
accuracy of the scale $\pm 1\ \mu\text{g}$
conditioning: 8. 24 h (20°C , rel.
humidity 50%)

- SMPS – Scanning Mobility Particle Sizer, TSI (DMA TSI 3071, CPC TSI 3025 A)
- NanoMet – System consisting of:
PAS – Photoelectric Aerosol Sensor (Eco Chem PAS 2000)
DC – Diffusion Charging Sensor (Matter Eng. LQ1-DC)
MD19 tuneable mini-diluter (Matter Eng. MD19-2E).

Particle Counts & Size Analysis:

As one of the principal objectives of the investigations, the measurements of particle size distributions and count concentrations were performed using:

The nanoparticles were determined at stationary engine operating conditions, each time before and after oxidation catalyst by switching the hot sampling device of NanoMet.

Elemental determinations

Differential elemental analysis was performed at EMPA Inorganic Analytical Laboratory. The emission aerosols sampled on quartz fibre filters (Munktell and Pallflex) were digested in acid mixtures using a *1200 Mega – High Performance Microwave Digestion Unit* (MSL GmbH). Multiple determinations have been carried out on punched filter segments. A detailed description of the digestion procedure can be found in [10][12][13]. Only suprapure and ultrapure quality acids (Merck) and high-purity water (18.2 M Ω cm), prepared by a Milli-Q Gradient A10 System (Millipore) were used for sample and standard preparation. Standards were prepared from single and multi-element standards all in ICP-quality (Merck and Alfa Aesar). The spectrometers used for determinations of the aqueous digestion solutions were a quadrupole inductively coupled plasma mass spectrometer (q-ICP-MS) *ELAN 6000* (Perkin Elmer/Sciex) and a high resolution magnetic sector field ICP-MS *ELEMENT II* (ThermoFinnigan) both operated under standard hot plasma conditions.

Elemental determinations in diesel samples have been performed with a radial inductively plasma optical emission spectrometer (ICP-OES) *VistaPro* (Varian). Chlorine and sulphur were directly determined using a Philips PW-1400 wavelength dispersive x-ray fluorescence spectrometer (WD-XRF).

Procedures of Investigation

All oils were investigated at steady state warm operation condition of the engine, at 5 operating points, Fig. 3.

Before change to another oil type the new oil was used to purge the engine without oil filter. The engine was operated for approx. 15 min. With the second definitive new oil filling a new oil filter was applied.

For particle size distribution measurements using SMPS always 3 scans with a total duration of 450 s were performed. In the same time the NanoMet signals were registered.

The particle sampling time on filters for gravimetry depended on the engine operating point and varied between 10 and 40 min, with the scope to obtain at least 1 mg of particle mass per filter for further analysis.

Fig. 3: Engine operating points in the test program

Oils and Tracing

Oils

The characteristics of the investigated lubrication oils are summarized in Table 1.

Property	Unit	Oil A 5W30	Oil B 15W40	Oil C
Viscosity kin 40°C	[mm ² /s]	70.57	108.9	94.92
Viscosity kin 100° C	[mm ² /s]	11.6	14	13.45
Viscosity index	[-]	159	135	142
Density @ 15°C ou 20°C	[g/ml]	0.8617	0.885	0.8392
Total Base Number	[mg KOH/g]		11.1	0.55
Pourpoint	[°C]	-54	-27	-54
Sulphated ash	[%]	1.52		
Sulfur	[%]	0.42	0.73	0.0005
Mg	[%]	0.04	0.0422	
Zn	[%]	0.14	0.1572	
Ca	[%]	0.33	0.3655	
P	[%]	0.13	0.1433	
Mo	[%]		0.0116	

The additive packages are a major source of elements, which generate the irreversible ashes in the DPF; ashes consisting mostly of sulphates, phosphates and oxides of Ca, Mg and Zn ^{[14][15]}.

Lowering the ash content of the lubrication oil without deteriorating the oil characteristics and durability is a difficult balance between the contradictory exigencies, which is a challenge joined with DPF technology.

Property	Unit	Oil D 15W40	Oil E 15W40	Oil F 5W30
Viscosity kin 40°C	[mm ² /s]	104.8	-	69.76
Viscosity kin 100° C	[mm ² /s]	14.22	13.98	11.8
Viscosity index	[-]	139	-	166
Density @ 15°C ou 20°C	[g/ml]	-	-	-
Total Base Number	[mg KOH/g]	13.5	8.4	5
Sulphated ash	[%]	1.8	1.077	0.47
Sulfur	[%]	0.65	0.336	0.2
Mg	[%]	0.001	0.001	0.022
Zn	[%]	0.127	0.12	0.105
Ca	[%]	0.473	0.263	0.06
P	[%]	0.116	0.111	0.095

Tracer Study

The tracer study was performed to investigate how much the generation of particles is influenced by the lubrication oil. Two oils, oil D 6500* 15W40 and oil F 2000 5W30, with different TBN and different S-content (Tab. 1) were applied. First, the tests were performed with the original oils. Secondly the tests were repeated with the doped oils.

The tracer was dosed with the tracer:oil volume ratio of 1:58. The tracer contained 1250 ppm lead Pb, 1250 ppm cadmium Cd, 1250 ppm barium Ba and 1250 ppm antimony Sb. Two elements which tend to form volatile compounds (Cd and Sb) as well as two elements which are known to show no such tendency (Ba, Pb) were selected. Additionally the omni-presence of the elements in the system (engine, oil, fuel) was taken into account.

Table 1: Characteristics of the studied lubrication oils

Different packages of additives are used to enable the lubricants to fulfil the various requirements.

Only the synthetic oil C was additive-free and could be used in the engine only for some hours. The oil C is a research oil, which is not commercially available and consists of a mixture of paraffin hydrocarbons between C12 and C26.

Additives protect the oil (antioxidants, antifoam) as well as the engine (anticorrosive, anti-wear) and control the long-life quality of the oil (detergent, dispersive, viscosity index, etc.).

*) the used numbers indicate ppm S in the oil

Results

Catalyst

Figure 4 illustrates some typical plots of exhaust gas components at constant speed, 2000 rpm, with one of the investigated oils:

- CO is reduced due to the conversion in the oxidation catalyst.
- The reduction of NO_x in the catalyst is only the artefact of the cold measurement, where the higher NO₂-share after the catalyst is more absorbed in the condensation water provoking more NO_x-losses; the hot measurement with a supplementary drying of the sampled probe can by a mayor part solve this problem. This was investigated at AFHB in connection with the VERT certification procedures of DPF systems, [6] [16]. The NO/NO₂ conversion in the oxidation catalyst has been controversially focused in the last years. This conversion is of primary importance for the functionality of certain after-treatment devises, like CRT, SCR, or DPNR, but can cause a higher NO₂-emission and a higher toxic exposition.
- PM increases at the lower load, because of the higher EGR-rate, which reaches in maximum approx. 25% [17].

The catalyst has also an effect on the nanoparticle emission:

- The SMPS particle size distribution spectra show after the catalyst lower count concentrations because of diffusion losses and partial oxidation of the precursor substances of condensates (SOF). A typical, very much repetitive example is given in Fig. 5.
- The NanoMet results, Fig. 6, show a reduction of nanoparticle emission in the catalyst.

Fig. 4: Emissions and fuel consumption at 2000rpm VW TDI 1896 cm³, with oil E 3360 *)

The signals of PAS and DC in this figure are converted to the values responding to the undiluted volume concentrations in the exhaust gas.

PAS (photoelectric aerosol sensor) is sensitive to the surface of particulates and to the chemical properties of the surface. It indicates the solid particles. DC (diffusion charging sensor) measures the total particle surface independent of the chemical properties. It indicates the solids and the condensates.

*) the used numbers indicate ppm S in the oil

Fig. 5: SMPS particle size distributions before/after catalyst, at 2000rpm VW TDI 1896cm³, with oil E 3360

Fig. 6: NanoMet results before/after catalyst, at 2000rpm VW TDI 1896 cm³, with oil E 3360

Figure 7 represents the reduction rates of nanoparticle counts (SMPS 20 - 300 nm) and surface (PAS / DC) in the oxidation catalyst with different lubrication oils at the same operating point 3000 rpm/90 Nm.

In this and in following figures the oils are listed in the sequence of increasing S-content (number ppm).

The reduction rates are in average: for SMPS & DC between 20 and 25 % and for PAS approx. 35 %.

One of the most important mechanisms is the store-release effect of the particles in the channels of the catalyst due to the diffusion filtration [18][19][20]. A part of the particles in the nanometer size range, adhere on the walls, agglomerates and can be released afterwards as bigger particles in micrometer range. These effects occur at different time in the different parts of the engine exhaust system [21][22] [23].

Another influence on the nanoparticles originates from the oxidation. On one hand the oxidation of SOF reduces the HC-condensates on the other hand the oxidation of SO₂ can increase the sulphate, hydrophilic condensates. The first effect is evidently predominant in the here reported results due to the used sulphur-free fuel.

Lubrication oils

Figures 8 and 9 represent the nanoparticle and particle mass emissions at different operating points with the different investigated lubrication oils. The oils are sorted in order of decreasing oil sulphur content. All represented parameters (SMPS, PAS, DC and PM) confirm at all operating conditions that the oils with the lowest S-content increase particle emissions, which is completely opposite to expectations.

Fig. 7: Reduction rate of nanoparticles R in the catalyst with different lubrication oils (engine at 3000 rpm/90 Nm)

*) the used numbers indicate ppm S in the oil

It can be concluded that the oil composition and the additive packages (except oil C) play an important role in the particle formation.

Regarding back to Fig. 7, it can be remarked that tendentially oils with higher emission values have also higher reduction rates. This is an indication for the fact that the differences of particle emissions for the different lubrication oils are due mostly to the differences of condensates (SOF).

- Influence of the oil on blow-by flow and blow-by composition.
- Acting of the oil at the cylinder walls during the combustion, oil evaporation, adsorption and desorption of fuel.
- Influence of the oil composition on the particle composition and on the condensation effects (speed of droplets growth).

To clarify all those effects and the possible secondary emissions a considerable further investigations are necessary.

Further investigations are also necessary to confirm the relationships of emissions of different oils at other operating conditions and on other engines. In the single measurements represented here the tendencies are confirmed by different parameters (PM, SMPS, PAS, DC) and by the similarity of these tendencies at all operating points.

Fig. 8: Nanoparticle emissions with different lubrication oils before catalyst, at 2000rpm

The observed emission differences are certainly an effect of combination of different influences, like:

- Acting of the oil with the piston rings and cylinder wall due to its viscosity.

Fig. 9: Particle mass emissions with different lubrication oils at 2000rpm (after catalyst)

These results confirm the higher particle mass and particle counts emissions of this engine, at part load operation, due to increased EGR (see Fig. 4).

Results of Tracer Study

The results of the tracer study should enlighten the contribution of lubrication oil on the formation of particulate matter in exhaust emissions. A main goal is an assessment of the lubrication oil contribution for different operation conditions of the engine. First results exemplarily for cadmium with and without tracer are presented in Figure 10. The tests with doped oil showed higher concentrations than the oils without tracer for all operation points. A similar trend was observed for the other elements in the tracer. A correlation between operating condition and the determinable amount of tracer, in the particulate matter is obvious. The lube oil has a considerable contribution to the PM, particularly at the part load operating conditions of the engine.

Further results will be published later.

Fig. 10: Cadmium concentration in sampled particle matter for oil D 6500 with and w/o tracer BP 1-5...operating points see Fig.3

Conclusions

Following conclusions can be pointed out:

- The oxidation catalyst reduces the nanoparticle emissions due to the diffusion-deposition and oxidation of SOF.
- Reduced sulphur and lower additive content of the lubrication oil don't necessarily reduce the particle emission of the engine.
- The complex influences of oil quality on the particle emissions offer several open questions for further research.
- The results of oil tracing show, that the lube oil has a considerable contribution to the PM, particularly at the part load operating conditions of the engine.
- An increased EGR rate at the part load of this engine provokes also increased emissions of particle mass and counts.

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Abbreviations

AFHB	...	Abgasprüfstelle der Fachhochschule, Biel CH (Lab. For Exhaust Gas Control, Univ. of Appl. Sciences, Biel-Bienne, Switzerland)
BUWAL	...	Bundesamt für Umwelt, Wald und Landschaft (Swiss EPA, FOEFL, SAEFL)
CO	...	carbon monoxide
CRT	...	continuously regenerating trap
DC	...	Diffusion Charging sensor
DPF	...	Diesel Particle Filter
DPNR	...	Diesel Particle & NOx Reduction
EGR	...	exhaust gas recirculation
EMPA	...	Swiss Federal Laboratories for Materials Testing and Research
FOEFL	...	Federal Office for Environment, Forests and Landscape (Swiss EPA, BUWAL)
HC	...	Hydrocarbons
INSOF	...	Insoluble Fraction
NanoMet	...	Thermomodulator + PAS + DC
NOx	...	Nitric oxides = NO + NO ₂
PAS	...	Photoelectric Aerosol Sensor
PM	...	Particulate Matter, Particle Mass
R	...	reduction rate $R = \Delta x / x_{b.cat}$
SAEFL	...	Swiss Agency for Environment, Forest and Landscape
SCR	...	selective catalytic reduction
SMPS	...	Scanning Mobility Particle Sizer
SOF	...	Soluble Organic Fraction
TTM	...	Technik Termische Maschinen
VERT	...	Verminderung der Emissionen von Realmaschinen im Tunnelbau (Swiss – Austrian – German project, DPF retrofitting in underground)
VFT	...	VERT Filter Test