DPF Systems for High Sulfur Fuels

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ABSTRACT

During the first decade of Diesel particle filter development and deployment in cars, trucks, buses and underground sites, DPF regeneration methods were engineered that were compatible with the then prevalent high sulfur content in the fuel > 2000 ppm. The mainly used methods were burners, electrical heaters, replaceable filters and non-precious metal fuel additives. Low sulfur Diesel fuel became only available from 1996 in Sweden, 1998 in Switzerland, and after 2000 everywhere in Europe. Thus, the deployment of precious metal catalytic converters was feasible both as original equipment and retrofitting of in-use engines. The so-called CRT particle filters using PGM-catalysis for providing NO₂ for low temperature regeneration became very successful wherever ULSD was available. However, in many applications, e.g. off-road and in the construction industry, Diesel engines continued to run on high sulfur fuel and in many emerging countries, even on-road Diesel fuel still contains between 1000 and 2000 ppm sulfur. These countries suffer very much from air pollution through increasing Diesel particle emissions and the high impact of black carbon particles on human health as well as on the global warming is worrying. Hence, the necessity for modern particle filters that are compatible with high sulfur content of the fuel. In the context of Chinese megalopolis, this paper reports investigation of a fuel which is typical for China

(containing > 1000 ppm sulfur) and compares results with European standard Diesel fuel. The test objects were two modern SiC particle filters, which were regenerated using different iron-based FBC. The combustion attributes of the soot were investigated by TGA and their EC/OC composition was examined. The results indicate that at the given test conditions the fuel sulfur does not significantly change the filters' physical and chemical properties. Neither the filter particle loading process nor the filter regeneration is noticeably different for the high sulfur test fuel compared to the ultra-low sulfur European fuel. Therefore VERT-verified iron-based FBC-type DPF can be used in countries where ULSD is not yet available.

INTRODUCTION

Before 1990, HDV Diesel fuel in most countries contained 2000 ppm sulfur. DPF compatible with high sulfur content fuel were developed beginning 1980. These developments were documented at the annual SAE Sessions on "Diesel Particulate Emission Control" [1] from 1982 onwards. Wall flow DPFs of this development period time were already very effective. The DPF deployment however, was limited to the retrofitting of in-use engines of public transport buses, subsequently construction machines and at underground sites. New engines

proved to be able to fulfill the legal PM mass-based emission limits of this period without a DPF and for this reason the DPF did not become yet a standard element of the OEM engine emission control technology at this time.

Low-sulfur fuels first became available from 1996 in Sweden. The main reason was the environmental impact on the Swedish lakes and forests due to SO₂ emission and sulfate formation, the so-called acid-rain formation. In other European countries and Switzerland the sulfur content fell stepwise from 2000 ppm to 500, to 350 ppm, to 50 ppm, and then in 2002 to 10 ppm.

This was the result of the Auto Oil Programs 1 and 2, mainly driven by two arguments: first, that the sulfur in the fuel was the major factor influencing particle formation. However, this holds only true when the overall particle mass PM is collected at low temperatures, irrespective of particle size and substance and also capturing the sulfate condensate plus bound water [2]. The solid content in this particle mass PM, consisting of insoluble ultrafine carcinogenic particles is independent of sulfur. The sulfate mass on the other side plus the bound water indeed constitute the volatile phase. The Worldwide Fuel Charter [4] confirmed that fact. Today including condensates in the PM-measurement is regarded to be a measuring artefact, resulting in far too low filter efficiencies [3] and misleading the emission technology development. This distinction between high toxic solid and low toxic volatile [21] however, was only recognized later and reflected in the European Directive EURO 6 [5] after the PMP instrumentation was implemented.

The second strong argument, which finally was decisive, was that high sulfur content in the fuel impeded the performance of precious metal catalysts. Low sulfur fuel cleared the way after 2000. It also triggered the introduction of NO₂ regenerating particle filter concepts like the Johnson Matthey CRT system and the Engelhard DPX system. The DPF retrofit market using these systems steadily grew, especially for public transportation. The inherent drawback of this concept was increased NO₂-emission which was underestimated and contributing to today's high NO₂ levels in street canyons having intense traffics [20]. Other filter systems using burner regeneration, base metal catalysis and fuel borne catalysts FBC were less successful because of higher complexity, lower performance or higher cost.

ULSD fuel also permitted the use of Diesel oxidation catalytic converters. Pt-based DOC became a standard component in European LD Diesel engines, permitting EGR for NO_x curtailment while diminishing CO and HC. Here, too, the increasing NO₂ emission was not limited. Grouping the oxides of nitrogen NO, NO₂ and N₂O which are very different with respect to their physics and chemistry as well as to toxic effects to human, non-weighed into a single "NO_x"-emission standard has blinded authority observance of emitted levels of NO₂ - in contrast to occupational health where this grouping was never accepted and consequently Pt-based DOC were banned in some countries from being used in mines.

The influence of the fuel sulfur on the Pt-based catalysts is well documented [6]. With the presence of sulfur and oxygen the catalytic converter favors the conversion of SO₂ → SO₃. This tenaciously inhibits other oxidation reactions, especially the reaction NO → NO₂, so that the CRT reaction finally ceases at low exhaust-gas temperatures. On the other hand, when ULSD is used the reaction NO → NO₂ is favored.

Introduction of low Sulfur fuel however, remained limited. Many developing and emerging countries still use fuels with > 1000 ppm S. Even most modern industrialized countries use this high-S cheaper fuel for construction machines, non-road and stationary engines, and locomotives. Fuels in ocean shipping have even higher S concentrations up to 4 %. Hence, emission control has been delayed in these sectors.

Nevertheless in most countries, the off-road sector contributes as much PM as the on-road sector, because off-road emission regulation has lagged behind on-road. Consequently, the use of particle filters would be much more cost effective for off-road application.

Particle filter technology for high polluting off-road engines, burning high sulfur fuels, was therefore studied to define best available retrofit-technology for these applications. Target areas of immediate interest are underground workplaces worldwide and construction machines in France, UK, Italy, China and other countries.

As a result of these considerations all systems using precious metal catalysts and catalytic combustion had to be excluded from these high sulfur fuel applications. Burner systems, base metal coatings and fuel borne catalysts were selected as promising candidates. This study started with the investigation of two FBC systems, both based on iron.

TEST ENGINE, LUBRICATION OIL AND TEST SETUP

Table 1. Test engine Liebherr D934 S

Manufacturer	Liebherr
Type	D934S
Cylinder volume	6.36 dm ³
Rated speed	2000 RPM
Rated power	105 kW
Max. Torque	680 Nm
Model	4 cylinder in-line
Combustion process	Direct injection
Injection pump	Bosch unit pump
Supercharging	TC / IC
Emission control	none
Design year	2005
Application	Construction machines

Table 2. Lubricant “Lubrizol Blue” research oil

Viscosity kin 40°C	ND	mm ² /s
Viscosity kin 100°C	13.98	mm ² /s
Viscosity index	ND	(--)
Density 20°C	ND	kg/m ³
Pour point	-25	°C
Flame point	ND	°C
TBN	8.4	mg KOH/g
Sulfur ashes	10 770	mg/kg
Sulfur	3 360	mg/kg
Magnesium	< 10	mg/kg
Zinc	1200	mg/kg
Calcium	2630	mg/kg
Phosphorus	ND	mg/kg

Tables 1 and 2 and Fig. 1 describe the test engine and the test set-up.

This construction engine has been repeatedly investigated during the VERT project. Several SAE papers report prior investigations on this engine [13, 14, 17]. All its attributes are well known and documented. In this study, the usual exhaust-gas instrumentation was enhanced with nanoparticle analysis. This comprises a SMPS system with subsequent CPC. The sampling is PMP compliant [7], i.e. the exhaust-gas sample is heated to 300 °C and diluted approx. 1:100. Hence, only solid particles are counted.

Fig. 1. Test setup [23]

TESTED FUELS

Table 3. Properties of tested fuels compared to on HSD Chinese Fuel

Type			Swiss market fuel	China market fuel	Test Fuel
Manufacturer			Shell	Sinopec	Shell
Property	Method	Unit	ULSD, SN 18 11 60-1-2009	HSD China Fuel Sample	HSD Formulated
Density (at 15°C)	EN ISO 12185	kg/m ³	825.2	848.6	841.8
Viscosity (at 40°C)	EN ISO 3104	mm ² /s	2.199	2.733	2.083
Cetane number	ISO 5165	-	49.9	45.8	45.3
Cetane index	ISO 4264	-	51.8	ND	ND
Sulfur content	EN ISO 20846	mg/kg	7.7	1223	1369
Cloud point	ISO 3015	°C	-11	ND	ND
Pour point (CFPP)	EN 116	°C	-23	ND	ND
Flash point	EN ISO 2719	°C	56.0	ND	ND
Heating value		MJ/kg	min.42.5	ND	ND
Aromatic hydrocarbons	ISO 3837	Vol %	max. 2	ND	ND
Boiling analysis (340°C)	EN ISO 3405	Vol %	96.1	91.1	94.4
Ash	EN ISO 6245	mg/kg	< 0.001	< 0.001	< 0.001
Fe of additized fuel	SPO 631	mg/kg	18.7 / 18.1	ND	18.5 / 18.1

The original Chinese fuel could not be exported from China to the Swiss engine lab. Hence the Chinese fuel was analyzed and Shell formulated a similar fuel, which was used in the tests reported here. The comparison shows that the Chinese Diesel

fuel is very similar to the European, except for the sulfur content, and is not contaminated with more organic or inorganic components.

TESTED PARTICLE FILTERS

Both tested filter systems are VERT certified [8].

Table 4. Tested particle filter systems

Manufacturer		Dinex A/S	Pirelli Eco Technology S.p.A.
Material		SiC	SiC
Porosity	[%]	45	42
Pore size	[µm]	15	20
Cell number	[CPSI]	150	180
Wall thickness	[mm]	0.7	0.43
Space velocity	[s ⁻¹]	30.2	22.8
Regeneration		FBC passive	FBC active
Manufacturer of FBC		Innospec	Pirelli Eco Technology
Specification		Satacen [®] 4	CAM-FBC active
FBC content max.		20 mg Fe / kg fuel	20 mg Fe / kg fuel

ANALYTICAL METHODS

GRANULARITY ANALYSIS

The size classification of the solid soot particles was determined using the SMPS procedure (Scanning Mobility Particle Sizer) [9]. The particle classification and particle counting was done after heating the gas sample to a temperature of 300°C and diluting approx. 1:100 (PMP compliant [7]). Thus only solid particles entered the instrumentation. The size classification is in very narrow bands, between 10 - 500 nm in 60 size classes. The defined particle size corresponds to the particle mobility diameter and is therefore representative for diffusion processes. This classification corresponds to deposition in the human respiratory system and penetration through the alveolar membrane [19]. The particle number concentration is quantified in a condensation core counter using the individual counting mode. The SMPS instrumentation from TSI with DMA 3081 and CPC 3772 were used.

THERMOGRAVIMETRIC ANALYSIS (TGA)

Soot was sampled at an average temperature of 320°C, diluted by the SmartSampler tunnel and thereby cooled to a temperature of 25-30°C at which temperature it was deposited on a quartz fiber filter for the thermogravimetric analysis.

The thermo-gravimetric procedure is well known and documented [10]. This investigation used the STA 409 CD instrument from Netzsch and a quadrupole mass spectrometer Aëolos, also from Netzsch, Germany. The measurements were done in pure oxygen atmosphere at a heating rate of 20°C/min. in the temperature range of 40°C - 800°C. The mass spectrometer was used to detect online the emission of the following substances:

- Water
- CO, CO₂,
- SO, SO₂

EC/OC ANALYSIS

The separation of the intercepted soot (again sampled on a quartz fiber filter) in elemental carbon EC and organic carbon OC indicates the character of the soot. It is an essential metric of the combustibility. Pure elemental carbon requires substantially higher combustion temperatures than a mixture with high organic content. The analysis was done using both the classical Coulometry (according to the German Standard VDI 2465) and also the NIOSH method 5040 [11]. The distinction EC/OC is also important in this investigation because in occupational health the Diesel particle emissions are usually evaluated according to its EC content [12].

RESULTS

RESULTS OF THE ENGINE TESTS

Comparison at Steady-State Point 1400 rpm and 340 Nm

At this medium load point, the engine is measured with respect to all limited emissions and thermodynamic data as usual. The main results are summarized in [Table 5](#).

With the exception of PM, the emissions are measured steady state as legally prescribed. The data are reported, as usual, energy specific in [g/kWh] terms. PM [g/kWh] could not be determined in these tests, because particle samples had to be collected for subsequent TGA- and EC/OC-analysis. The reported PM [g/filter] data correspond to the loading cycle as described in [Fig.4](#). There is no significant disparity in the [Table 5](#) gaseous emissions and in the fuel consumption. The particle mass PM is higher for the tests with iron additive, as expected.

Particle Size Analysis in the Raw Gas (Without DPF)

[Figures 2](#) and [3](#) summarize size analysis with all 4 fuels at the operating point at 1400 RPM and 340 Nm.

Table 5. Limited emissions CO, HC, NOx, fuel consumption and PM

	CO	HC	NOx	Fuel Consumption	PM
	[g/kWh]	[g/kWh]	[g/kWh]	[g/kWh]	[g/ filter]
ULSD	0.62	0.25	6.79	227.4	2.2
HSD	0.69	0.24	7.49	227.9	2.17
ULSD + FBC	0.65	0.26	6.82	230.3	2.6
HSD + FBC	0.65	0.26	7.28	229.0	2.41

Fig. 2. Size distribution comparing the 4 fuel variants with gas sample pre-heating to 300°C, as per PMP [7]

The size distribution reveals almost no difference for the 4 fuel variants. The iron additive causes the expected increase of particle count in the range 10-25 nm. These are ultra fine iron oxide clusters formed after combustion of the metal organic additive substances. These very fine particles result in a large specific surface that promotes catalytic effects during the filter regeneration. VERT certified DPF systems very efficiently intercept these fine FBC oxide particles as they remove all solid particles including metal oxide particles from engine wear and lubrication oil by over 99 % [23] to an extent that they are usually not detectable in the size spectrum after the DPF [13]. The filtration attributes of these two VERT-certified DPF are well documented [8]. Hence, size measurements were not done after the DPF.

The measurements were repeated with sample heating to only 100°C.

This investigation was done to observe the condensate formation. 100°C is far below the condensation point of sulfuric acid vapors. Sulfur contained in exhaust-gas, at temperatures below 300°C, usually generates a distinct bimodal size distribution with exceptionally high peaks [14]. This investigation surprisingly found no condensate in the cool sample. With Pt-catalysis, sulfates would be formed according to the processes: $\text{SO}_2 + \frac{1}{2} \text{O}_2 \rightarrow \text{SO}_3$ and with the water present in the exhaust: $\text{SO}_2 + 8\text{H}_2\text{O} + \frac{1}{2} \text{O}_2 \rightarrow \text{H}_2\text{SO}_4 + 7 \text{H}_2\text{O}$, which explains the high contribution to PM mass. Apparently the exhaust-gas did not contain SO_3 and hence sulfuric acid was not formed. The sulfur in the fuel was obviously completely converted to SO_2 and passed the system as gas, without forming sulfates.

Fig. 3. Size distribution comparing the 4 fuel variants with the gas sample pre-heated to only 100°C in order to provoke sulfuric condensates

Filter Loading and Regeneration

The filters became soot-loaded in a dynamic cycle as illustrated in Figure 4. Soot was generated in rapid acceleration cycles from idling until full load, thus avoiding the temperature rising to values at which the FBC doped fuel would trigger filter regeneration. The aim was to attain the highest possible filter loading to subsequently test the regeneration response.

Fig. 4. Filter loading cycle

The exhaust backpressure rise, during filter loading, was not uniform for all fuels. This can be explained as follows: the test started with a new filter, which after regeneration still had some residual soot load. The second loading cycle thus has a distinctly different character and cannot be compared with the first. This will be further investigated at a later stage of this investigation.

Regeneration Response

The filter loaded with soot in the described cycle, was regenerated according to the Swiss standard test protocol SNR 277 205 [15]. This test was only performed for the two FBC doped fuels. The results are shown in Fig. 5 and Fig. 6.

The regeneration begins below 340°C exhaust-gas temperature. The load is increased every 20 minutes until full load. Plotted are the temperatures T7 before DPF and T8 after DPF, torque, pressure loss across the filter Δp_7 and the particle emission in the CPC count.

The results (for two different DPF, two different additives but both iron-based and both 20 ppm dosage) show that, already at the first operating point, the back-pressure decreased despite increasing soot load and increasing volume throughput. Already at this 340°C temperature, the balance-point is passed.

Fig. 5. Regeneration of the Dinex filter. ULSD and HSD, both with FBC

Fig. 6. Regeneration of the Pirelli filter. ULSD and HSD, both with FBC

Increasing the temperature steepens the regeneration gradient until termination of the test after running 20 minutes at full load. The regeneration is obviously not yet complete, as evident in the gradients. Hence, the next test starts with slightly different initial conditions. Despite this imperfection of the test procedure, there is almost no evident difference between the two fuels.

The difference of the two CPC plots seems somehow striking: whereas the Dinex is in the range of 10^2 particles per cc, Pirelli is in the range of 10^5 . This should be compared to the raw emission (See Fig 2) which is in the range of 10^7 . In other words: while Dinex shows filtration efficiencies of $> 99.99\%$ in this test the Pirelli filter “only” shows 99% . The difference in pore size and wall thickness may contribute to this difference but also slightly different soot layer thickness could explain most of it. It should be emphasized here that these VERT-certified filters keep filtration of solid ultrafine particles to a level of $> 99\%$ even during regeneration.

THERMOGRAVIMETRIC ANALYSIS

The entire investigation comprised four fuel variants. Fig. 7 with thermo-gravimetric data, coupled with simultaneous MS-detection of volatile combustion products (H_2O , CO , CO_2 , SO , SO_2) only compares ULSD to HSD, both with FBC. The grey line pertains to data for ULSD. The black line pertains to data for HSD, which is the test fuel having high sulfur content. Charted as a function of the increasing temperature are: mass loss and ion current, water vapor, CO , CO_2 , SO , SO_2

Ion-currents recorded for the observed volatile decomposition products are proportional to the volume portion of the decomposition product in the TG exhaust. The lower figure shows the sulfur decomposition products at a higher resolution.

The response is surprisingly similar for the low-sulfur and the high-sulfur fuel. The similarity prevails for the emission of water vapor and the first drying phase, at temperatures above $100^\circ C$, and also for the initiation of soot combustion evidenced by spikes of CO and CO_2 . Very remarkable is the response of gaseous sulfur emissions SO and SO_2 . These emissions are exceptionally low, in the entire temperature range, including the gradual increase during soot combustion. Noteworthy is that combustion of the high sulfur fuel soot does not produce more SO and SO_2 emissions. This investigation thus confirms that the high sulfur content does not proportionally deposit on the soot, but obviously exits the system as gaseous SO_2 .

Figure 8 is another interesting comparison. It shows the thermo-gravimetric analysis for the effects of the fuel additive on the regeneration.

Evidently, the FBC triggers the start of regeneration at about $340^\circ C$, which is 160 K below the temperature the soot is starting to burn without FBC support and thereby enables faster and more complete filter regeneration, which however, is not visible in this diagram.

Fig. 7. Comparison of the TG-MS data recorded for ULSD and HSD; both with 20 ppm iron additive

Fig. 8. TGA analysis for the ULSD fuel with and without iron FBC

EC/OC

During filter loading according to Fig.4, samples are collected on quartz filters. These samples are analyzed using the Coulometry procedure VDI 2465 as well as the NIOSH procedure [11]. Table 6 summarizes the results:

Table 6. EC/OC analysis of soot samples from filter loading test

[µg/ filter]	Coulometry VDI 2465			NIOSH 5040 [11]		
	OC	EC	EC/TC	OC	EC	EC/TC
ULSD	212	1838	89.7%	205	1905	90.3%
HSD	236	1750	88.1%	238	1849	88.6%
ULSD + FBC	307	2193	87.7%	409	2157	84.1%
HSD + FBC	279	1893	87.1%	317	1882	82.5%
Blind value	44	26	-	50	4	-

The data listed for OC and EC are the absolute mass per filter sample. This mass naturally differs from case to case. These absolute values are therefore not mutually comparable. Instead, the ratio of elemental carbon EC to total carbon TC = EC + OC is compared for both measurement procedures. Despite the inevitable measurement variability, the results are very uniform. Hence, there is no identifiable disparity between the fuel variants. The HSD data tends to a slightly lower EC/TC ratio, but the differences are rather insignificant.

CONCLUSION

This investigation compared 2 DPF-systems using iron based FBC-regeneration with European Diesel fuel (sulfur content < 10 ppm) and high sulfur test fuel (sulfur content 1369 ppm) as it is typically used in China. No significant differences were found in the limited emissions CO, HC, NOx and PM, the EC/OC ratio of the soot, in the characteristics of residual soot combustion (TGA) and the regeneration response. Evidently, the FBC iron-oxide enables that the sulfur (both in the fuel and in the lube oil) entering combustion subsequently exits as gaseous SO₂. No sulfate is formed and thus the particle formation is unaffected. In contrast to this: whereas when a precious metal catalyst is deployed with high sulfur fuel, SO₂ can be converted to SO₃ as published in [16], [17], [18] and [20] - thus sulfate is formed, which increases the particle mass and inhibits filter regeneration. Consequently, precious metal DOC and Pt-coated DPF are unsuitable for HSD fuels. But particle filter systems without catalytic attributes, or with base metal catalysts, can perfectly cope with very high sulfur fuel content. The soot particles are efficiently filtered, without undesirable side-effects, and the filters are effectively

regenerated. This ensures durable and dependable operation. These conclusions are of course limited to short term effects and to the operation profiles used during this investigation. Further investigations will be required.

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ACRONYMS AND ABBREVIATIONS

AFHB

University of Applied Sciences, Biel, Switzerland

BM

Base Metal

CPC

Condensation Particle Counter

CPSI

Cells per square inch

CRT

Continuously Regenerating Trap (trademark Johnson Matthey)

DC

Diffusion Charger

DOC

Diesel Oxidation Catalyst

DMA

Differential Mobility Analyser

DPF

Diesel Particle Filter

DPX

Catalyst coated DPF (trademark Engelhard)

EC

Elemental Carbon

EGR

Exhaust Gas Recirculation

EMPA

Eidgenössische Material Forschungs- und Prüfanstalt, Dübendorf, Switzerland

EN, ISO, SN

Standards: European Norm, International Standard, Swiss Norm

FBC

Fuel Borne Catalyst

HSD

High sulfur diesel

LD

Light duty

MS

Mass spectrometry

nm

Nanometer = 10^{-9} m; νάννος (greek) = the dwarf

NIOSH

National Institute for Occupational Safety and Health

NO

Nitrogen monoxide

NO₂

Nitrogen dioxide

OC

Organic carbon

PAS

Photoelectric Aerosol Sensor

PGM

Platinum Group Metals

PM

Particulate matter

PMP

Particle Measurement Program

ppm

Parts per million

S

Sulfur

SO, SO₂

Sulfur oxides

SMPS

Scanning Mobility Particle Sizer (Instruments TSI, USA).

TGA

Thermogravimetric analysis

TC

Total Carbon = EC + OC

ThC

Thermo-Conditioning = preheating of the gas sample line

TTM

Technik Thermische Maschinen, Consulting Switzerland

ULSD

Ultra low Sulfur Diesel

VDI

Association of German Engineers

VEMC

Vehicle Emission Management Center, Beijing

VERT[®]

Verification of Emission Reduction Technologies, a trade mark

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