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Ash generation options for accelerated testing of marine diesel particulate filters

Exhaust Gas Aftertreatment Solutions & CCS

Päivi Aakko-Saksa, VTT Technical Research Centre of Finland

Kati Lehtoranta, VTT Technical Research Centre of Finland
Hannu Vesala, VTT Technical Research Centre of Finland
Anssi Järvinen, VTT Technical Research Centre of Finland
Nahil Serhan, Wärtsilä
Jan Torkulla, Wärtsilä

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ABSTRACT

Particulate matter (PM) emissions, comprising black carbon (BC), organic fraction and sulfates, pose significant health risks, and the shipping sector substantially contributes to the PM levels, especially in coastal cities. Notably, air pollution remains the main environmental cause of premature deaths. Additionally to the health risks, BC emissions from shipping strengthen the global warming effects through deposition of BC on ice and snow. Practically, north of 70° latitude, shipping is the predominant source of BC emissions. The role of ship emissions is also increasing with the anticipated rise in commercial shipping, particularly in the Arctic. Since 2011, the International Maritime Organization (IMO) has worked on BC emissions from shipping.

Diesel particulate filters (DPF) effectively reduce BC and PM emissions. Although DPF technology is well-established for vehicles and non-road mobile machinery, its adoption for marine diesel engines presents unique challenges related to the properties of marine fuels and engine oils (sulfur content, ash), and the regeneration process of the DPF. Ash plays a crucial role, as soot can be removed from the DPF through regeneration, but the accumulated ash cannot be cleaned simultaneously.

To develop and verify the performance of marine DPFs, an accelerated ash accumulation method is necessary. We investigated four different ash generation methods: 1) a burner type ash generator, 2) a modern diesel engine, 3) a robust diesel generator, and 4) injection of high-ash engine oil into the engine's intake air. Additionally, we refined a methodology to screen the ash content of engine exhaust.

1 INTRODUCTION

Particulate matter (PM) emissions from the shipping sector are harmful to health and environment, and notably, the black carbon (BC) deposition on ice and snow in the Arctic strengthens the global warming. Since 2011, the International Maritime Organisation (IMO) has worked on BC emissions from shipping. In the Sulfur Emission Control Areas (SECA) marine fuels may contain up to 0.1% of sulphur, while the global sulphur of 0.5% was set by the IMO in 2020. In July 2024, maximum limit for marine fuel viscosity was introduced in the Arctic. However, even distillate and hybrid marine fuels contain substantially sulphur, heavy organic compounds and metals when compared with road transport fuels, and so are these constituents also emitted with ship PM [1] [2].

Reduction of PM and BC emissions from ships with the diesel particulate filter (DPF), a well-known technology for vehicles and non-road mobile machinery, is not straight-forward due to the quality of marine fuels and engine oils, particularly their ash content. Soot can be removed from the DPF through regeneration, but the accumulated ash is not cleaned simultaneously. DPFs for marine diesel engines have been introduced since 2005 [3]. Recently, Lehtoranta et al. [4] reported of the results from an EU stage V certified medium-speed engine (2000 kW, 1000 rpm, 8-cylinder in-line, Anglo Belgian Corporation, ABC) equipped with the DPF and the Selective Catalytic Reduction (SCR) systems, which do not contain noble metals enabling the usage of fuels with sulfur levels up to 0.5%. PN and BC filtration efficiencies were reportedly over 98%. Burners remove soot collected on the DPF regularly by increasing the temperature sufficiently for soot oxidation. These DPF systems are reported to operate over 6000 hours before need for cleaning with the SECA-compliant distillate fuel [4][5].

Ash content of fuel and lubricating oil crucially affect the marine DPF performance. There is no standardised procedure to study effectiveness and operation of the marine DPF or how to generate ash to mimic that originating from marine fuels and high-ash lubricating oils. Interestingly, a procedure for Diesel Accelerated Aftertreatment Aging (DAAAC) extensively studied [6,7] was recently published for high-speed diesel engines using road diesel fuels with low sulphur content in the Code of Federal Regulations [8]. The DAAAC protocol defines the target oil exposure rate during accelerated aging as 10 times the field average oil consumption rate. The protocol defines two pathways for oil exposure a) bulk pathway from engine and b) the volatile introduction pathway with oil doped in the fuel or burner. The volatile pathway

should represent 10-30% of the total, while the rest is introduced through bulk pathway. The DAAAC defines also fuel sulfur exposure targets. The DAAAC recommend the preference in the bulk pathway is on installing the second compression ring inverted (upside down) on one or more of the cylinders or modifying the oil control rings to reduce the spring tension on the oil control ring or to create small notches or gaps in the top portion of the oil control rings that contact the cylinder liner. The DAAAC requires the oil consumption to be monitored periodically by draining and weighing, even if oil consumption was also continuously monitored. The secondary tracking of oil exposure can be done with the DPF, by using soot free DPF weights to determine the ash loading, which should be 55-70% of the mass predicted using the measured oil consumption and its ash concentration.

In order to produce ash for the marine DPF studies, methods are needed to generate elevated levels of ash in the exhaust in small scale. However, the ash content of the exhaust should not be unrealistically high to prevent an uneven ash distribution in the DPF and blocked spots that would not realise in the normal operation. This aspect is discussed in our study based on the observations and findings in the experimental work.

We developed three different ash generation methods for the performance testing of the marine DPFs: 1) a burner type ash generator, in which a marine fuel doped with high ash lubricating oil was used; 2) marine fuel doped with the high-ash lubricating oil was used in a modern non-road diesel engine and in a robust diesel generator; 3) injection of high-ash lubricating oil into the engine's intake air. Additionally, we refined a methodology to screen the ash content of engine exhaust. The ash generation methods were explored with the marine DPF samples designed for ships operating with fuels containing up to 0.1 wt% sulphur. This study contributes in the development of aftertreatment technologies to alleviate climatic and health effects associated to ship particle emissions.

2 METHODS

2.1 Fuels, oils, engines, DPFs

DMA and DMB fuel qualities were used in this study (Table 1). Characteristics of the high-ash lubricating oil, Mobilgard™ M440 (abbreviated M440 hereinafter), used in the ash generation methods is shown in Table 2.

Table 1. Characteristics of the DMB fuel according to the refinery certificate.

Characteristics	DMA	DMB ¹
Viscosity at 40 °C, mm ² /s	8.5	7.5
Density at 15 °C, kg/m ³	877	876
Sulphur content, wt%	0.09	0.02
Net heat of combustion, MJ/kg	42.4	42.5
Ash, wt% ²	0.002	<0.001
Carbon content, wt%	87	86.9

¹Refinery certificate for other fuel properties than ash. ² Ash was analysed by ISO 6245 without a step to produce sulfated ash.

Table 2. Characteristics of high-ash lubricating oil.

Characteristics	Mobilgard™ M440
Flash point, °C	242
Kinematic viscosity at 100 °C, mm ² /s	14
Pour point, °C	-6
Specific gravity 15°C/15°C	0.915
Sulphated ash, wt% ¹	5
Total base number, mgKOH/g	40

¹Method with a step to produce sulfated ash for lubricating oils containing ash-forming additives.

The engine to produce exhaust for the testing was a high-speed non-road AGCO 44 AWIC diesel engine (DE) (Table 1). Additionally, the robust diesel generator (DG) was used in the second phase of fuel doping study.

Engine oil used in the DE was Shell Rimula 10W30. In the data sheet of Shell Rimula 10W30, its content of sulphated ash is in maximum 1.5%.

Table 3. Characteristics of AGCO 44 AWIC diesel engine and the DG.

Characteristics	Diesel engine (DE)	Diesel generator (DG)
Nominal power, kW	94 @2200 min ⁻¹	4.2
Nominal torque, Nm	550 @1500 min ⁻¹	-
Number of cylinders	4	1
Displacement, L	4.4	0.455
Compression ratio	16.5	-
Fuel system	Common-rail, turbocharged, intercooled	Naturally aspirated, direct injection, mechanical

The DPF elements studied were provided by Wärtsilä.

2.2 Three ash generation methods

The test setup consisted of an engine, measurement devices, VTT's test bench facility and three ash generation methods (Figure 1). In the test bench, the exhaust flow through the DPF can be adjusted from 25-120 kg/h and the exhaust temperature range is up to approximately 550 °C.

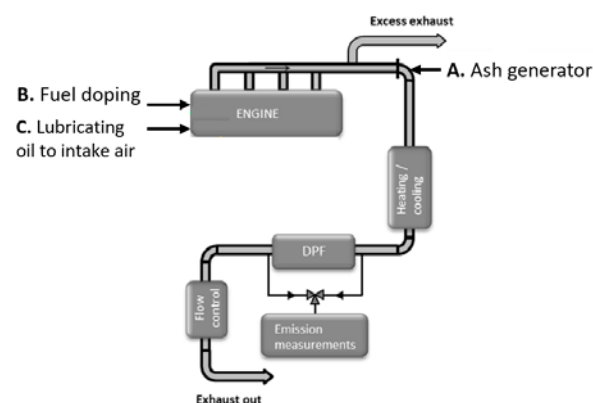


Figure 1. Simplified graphic of the test set-up with the three ash generation methods, which were used separately.

A. The ash generator was developed from a commercial boiler and burner. A small power boiler (Jäspi ECO 17 LUX-T) used typically in detached houses was selected as a basis of the system. An oil burner (Oilon BF 1 FUV HC) was selected for the system. The burner was equipped with a small oil nozzle providing fuel oil flow of 1.3 kg/h. The burner is equipped with an internal oil pump. An oil filter and an air removal system were installed in the oil feed line.

The ash generator required auxiliary systems to ensure reliable operation in which the burner is constantly on. A recirculation pump was installed to sustain constant water flow from the boiler to a heat exchanger and back to the boiler. Tap water was used to remove the heat in the exchanger. The flow of cool tap water was controlled with a thermostat, which measured the water temperature in the boiler and adjusted flow of the cooling tap water. The boiler was also equipped with a pressure relief valve and a volume compensator. The boiler has also an electric heater, which can be used to maintain temperature in the boiler and prevent condensation.

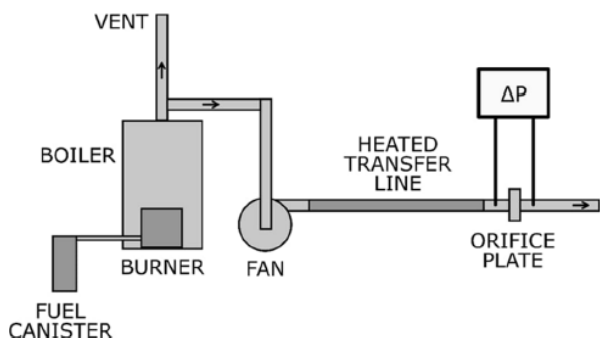


Figure 2. Schematic of the ash generator with additional components used in the measurements.

B. Fuel doping. Fuel was doped with marine diesel engine lubricating oil, ExxonMobil Mobilgard™ M440 (Table 2). Fuel blends studied contained lubricating oil concentrations between 0.2-2 wt%. Doping the fuel with lubricating oil required careful operation. First, the desired lubricating oil amount was weighed and then it was mixed to the fuel tank with Flux type pump-mixer. After the mixing was ready, the doped fuel was normally delivered to the engine.

C. Intake air injection. High ash lubricating oil injection in intake air was one way to introduce the lubricating oil to the engine combustion. For this method a Gilson Miniplus pump was employed to insert either pure lube or a mix of lube and fuel to the intake air. To control the amount of the injected lubricating oil, a mass flow meter was used, and the inserted lube canister was also placed on a balance. The lubricating oil injection rates studied were between 1-2 ml/min.

2.3 Emission measurements

Gaseous emissions, including NO_x , NO_2 , N_2O , NH_3 , CO_2 , CO and THC , were measured on-line at 20 seconds intervals using the Fourier transformation infrared (FTIR) equipment (Gasmeter DX-4000). Sample for FTIR was wet raw exhaust gas at temperature of 180 °C.

Continuous soot measurement was done with AVL Micro Soot Sensor (MSS, photoacoustic method). The results obtained by AVL MSS represent black carbon concentrations in exhaust.

Continuous PN measurement was measuring non-volatile, solid particle number emissions with Dekati DEED and Airmodus A23 CPC system according to EU Stage V legislation. Before DEED, the sample gas was diluted with Dekati eDiluter. Dilution air for eDiluter was dried, filtered and heated.

To measure total particulate matter (PM), The AVL Smart Sampler (SPC) was used for partial flow dilution and sampling of total PM. Partial flow dilution system combined with gravimetric sampling of exhaust particulates is a standardised procedure (ISO 8178). Pallflex TX40 filters were used for PM collections.

2.4 Ash determination methods

There is no direct methodology for measuring ash concentrations of the exhaust. The primary method to determine ash accumulated in DPF was weighing the soot-free DPF before and after the measurement period and periodically. Besides DPF weighing, ash content of PM was screened by subtracting total carbon (TC) from total PM mass. This method may overestimate the exhaust ash concentrations, since besides carbon and ash, PM may contain water combined with sulphates [9][10].

TC content of PM was analysed quantitatively by thermal-optical analysis (TOA, instrument Sunset Laboratories Inc's model 4L). PM was collected on quartz filters (Pallflex Tissuquartz), pre-cleaned (850 °C, two hours) and stabilised for several days. PM collection time was 600 seconds for TC analyses, while shorter for the elemental (EC) and organic carbon (OC) analyses. The protocol used was EUSAAR2 protocol, EN 16909.

Concentrations are presented in standard conditions of 273.15 K and 100 kPa, abbreviated as Sm^3 .

3 RESULTS

3.1.1 Burner based ash generator

DMB was doped with 20%, 10%, 5% and 1.25% of lubricating oil (M440) in the development phase of the burner-type ash generator. In the performance tests of the DPFs, DMB doped to concentrations to 5% and 1.25% of lubricating oil were used.

Ash concentration level from ash generator were at expected level. In the DPF performance tests, ash concentration of exhaust was on average 27 mg/Sm^3 with an ash generator (Figure 3). The ash level obtained with an ash generator with the selected fuel doping level was more than ten times higher than the natural ash content in the exhaust from this diesel engine with the fuel and lubricating oil used. Soot level from the ash generator fluctuated substantially with this concept.

3.2 Lubricating oil injection to intake air

Lubricating oil injection to intake air resulted in relatively low ash concentrations. Ash concentration additional to theoretical natural ash concentration with this setup was at the level of 2

mg/Sm³. Potentially, a part of lubricating oil did not reach the combustion chamber.

3.3 Doping fuel with high-ash lubricating oil

Several concentrations of the lubricating oil in the DMA and DMB fuels were studied. The highest concentration of the high-ash lubricating oil, 2% in DMA, led to engine failure. Lower concentrations of the high-ash lubricating oil in fuel led to undesired operation of the diesel engine, particularly to an increase in soot levels, which prevented the continuation of the measurements. Upgrading the standard injector configuration to larger injectors enabled the engine to run for a longer time period although soot levels were still fluctuating. The soot concentration returned to the original level after changing back to undoped fuel enabling successful performance testing of the DPFs. Examples of the exhaust ash concentrations with doped fuels are shown in Figure 3.

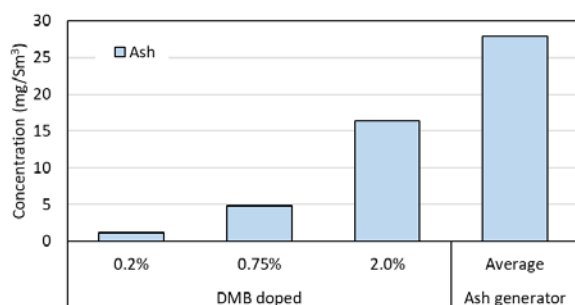


Figure 3. Ash concentrations in the exhaust when using fuel doped with 0.2-2% of M440 lubricating oil and when using a burner-type ash generator.

3.4 Discussion on ash generation methods and a diesel generator pathway

The theoretical ash concentrations expected in the exhaust originating naturally from fuel and lubricating oil, and from doping of fuel with lubricating oil and by injection of the lubricating oil in the intake air are presented in Table 4.

The natural ash accumulated in the DPF from the DMB fuel could be as high as 1.15 mg/Sm³ provided that the fuel contained maximum ash concentration of 0.010 wt% allowed by the ISO 8217 standard. However, the DMB fuel in our study was practically ash-free and lubricating oil in the engine was also of low-ash quality leading to only 0.06 mg/Sm³ theoretical ash concentration in the exhaust. Since naturally accumulated ash concentration in the exhaust was low, the ash accumulated was predominantly originating from the studied ash generation methods. Notably, the natural ash concentration is below the detection limit of the TOA-based ash screening method.

Ash concentrations measured in the exhaust when using three ash generation methods were as expected.

The highest ash concentrations were achieved by the burner-type ash generator in which high concentrations of the high-ash lubricating oil in fuel can be used. The burner-type ash generator enabled flexible adjustment of the ash generation levels, although this was not straightforward due to fluctuation of the soot emissions from burner and need for frequent cleaning of burner. Furthermore, PM may be different with the burner-type ash generator than with the diesel engine combustion process. Ash in the exhaust is produced in the cylinder during the combustion process under high temperature and pressure conditions. In contrast, burner-type combustion does not have the same pressure conditions, which may result in significant variance in ash morphology. Hence, other ash generation options were also studied.

Injection of the lubricating oil in intake air of non-road diesel engine was technically successful, however, the lubricating oil seemed not to reach completely the combustion chamber or, if it did, it was not being completely burned. The ash content in lubricating oil is primarily due to the inorganic additives, such as detergents and anti-wear agents, which contain metal compounds. When the oil is combusted, these metal compounds form metal oxides, which are referred to as ash. If the oil is not combusted, these inorganic compounds remain dissolved or suspended in the oil and do not form solid ash. This pathway was not selected for use in the performance tests of the DPF samples.

Fuel doping with lubricating oil was an interesting method to generate ash for the DPF studies. However, the high-speed non-road diesel engine used in the study is not designed for the use of poor-quality fuels and the increased soot levels, even at low levels of fuel doping, indicated problems in the fuel injection and combustion process. For the DPF performance tests, stable soot emission level is needed. Switching to clean fuel enabled achieving a stable soot emission level; however, the fuel change was slow due to the large intermediate fuel tank used, making control of the performance testing challenging.

Another approach to adopt fuel doping principle was using the robust small diesel generator (DG) to overcome challenges faced with fuel doping for high-speed diesel engine. Small DG proved to tolerate more high-ash lubricating oil dopant in the DMB than the diesel engine. With the DG, fuel changes were flexible, allowing a stable soot level to be reached relatively quickly, which enabled controlled DPF performance tests. Combination of

the DG and doped fuel was deemed to be the most flexible of the studied ash generation methods for the DPF performance tests. Even for the robust diesel generator, fuel doping levels are limited to protect the engine and the resulted ash concentrations are lower than those obtained by the ash generator.

The burner-type ash generation method would offer the most flexible choice for the desired ash concentrations in the exhaust, if the PM characteristics would be similar to those from the diesel engines. Characteristics of the PM obtained with the smaller engines and marine engines may also be different from each other, however, in both cases marine fuels and lubricating oil are used in the diesel combustion.

In this study, high-ash lubricating oil was the source of the ash. However, the ash-forming elements in the marine fuels and lubricating oils are different. In our earlier study [11], PM samples and lubricating oil contained substantial amounts of calcium (Ca), while it was less present in marine fuels. Vanadium (V), on the other hand, was present considerably in fuel and PM samples, while not in the lubricating oil. Simulating the ash generation only with lubricating oil doping does not reveal the influence of possible ash generating elements of fuel. However, in our studies the DMB batches contained only a minor amount of ash and hence the role of lubricating oil is pronounced more than the role of fuel for the ash accumulation in the DPF.

Table 4. Theoretical¹ ash concentration of the exhaust naturally, with mixing lube in fuel (doping) and injection of lube in intake air.

Characteristics	Naturally DMB (low/high)	Naturally 10W30/M440	Doping 1 lube M440	Doping 2 lube M440	Doping 3 lube M440	Injection in intake air ² M440
Ash content of fuel and lube, wt%	0.0005/0.010	0.75/2.5	2.5	2.5	2.5	2.5
Doping level of fuel, wt%	-/-	-	0.5	0.75	2	-
Theoretical ash conc. in exhaust, mg/Sm ³	0.06/1.15	0.05/0.15	3.5	5.3	14.0	4.3

¹ Assuming fuel consumption of 11.5 kg/h and lube consumption of 0.15 g/kWh at 40 kW

² Injection rate of 1.3 mL/min.

3.5 DPF performance tests

In the DPF performance tests, sequential phases of soot accumulations, regenerations and ash accumulations were carried out. High soot modes were needed for accelerated accumulation of soot. Low soot mode was used when the DPF performance was studied with different Gas Hourly Space Velocities (GHSV). High soot concentrations upstream DPF were achieved by decreasing the rail pressure from the OEM settings with diesel engine. With diesel generator, soot level was adjusted by load setting. With DMB fuel, diesel engine emitted relatively constant high and low soot levels, while with doped DMB, soot level fluctuated. Total exhaust composition may affect the DPF performance, hence also NO_x and total hydrocarbon (THC) concentrations, were studied.

Soot and ash accumulation, regeneration temperature and DPF reduction efficiencies of soot and non-volatile particles were monitored. Here, some examples of the performance tests of the DPF samples are shown.

Ash accumulated rapidly with burner-type ash generator and smoothly with other methodologies (Figure 4). With the burner, ash accumulation in the

DPF over the testing period was 24-38 g based on the weighing of the DPF before and after testing. Calculated ash accumulation was 22 g, respectively. Higher result from weighing than from calculation could be explained for example by accumulation of some heavy organic material in the DPF. With lubricating oil injection, ash accumulated in DPF according to the weighing was 15 g, which was in the same order of magnitude as that based on calculations, 19.5 g. In the calculations, assumed ash concentration of 2.05 mg/Sm³ was used based on PM and TC results.

Generally, the DPF samples tested reduced efficiently soot and particle number concentrations even when ash and soot loading was increased. With the burner-based ash generation method, particulate matter contained substantial share of elemental and organic carbon, both of which were efficiently reduced by the DPF. Engine out soot emission was on average 7.8/0.8 mg/kWh upstream and 0.03/0.003 mg/kWh downstream DPF in the high-soot and low-soot modes (ISO 8178).

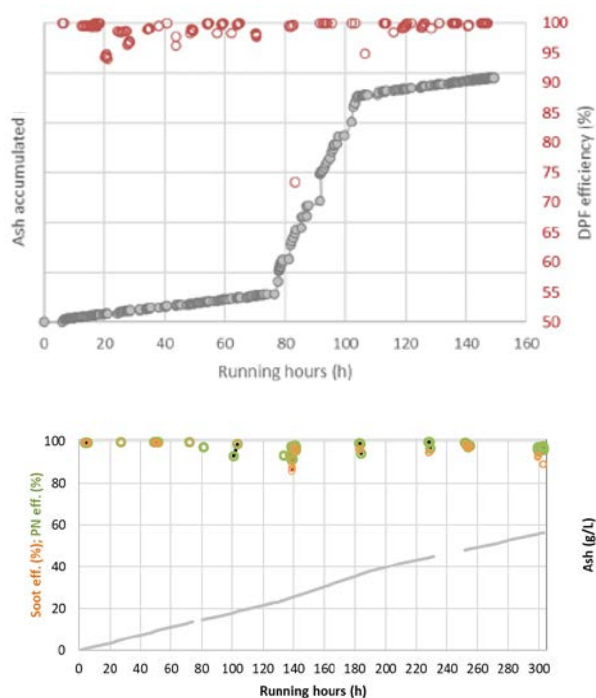


Figure 4. Ash accumulated rapidly with burner-type ash generator (upper) and smoothly with other methodologies (lower).

Pressure difference (dP) over the DPF clearly increased when ash and soot accumulated to DPF, especially at higher gas hourly space velocity (GHSV) levels (Figure 5).

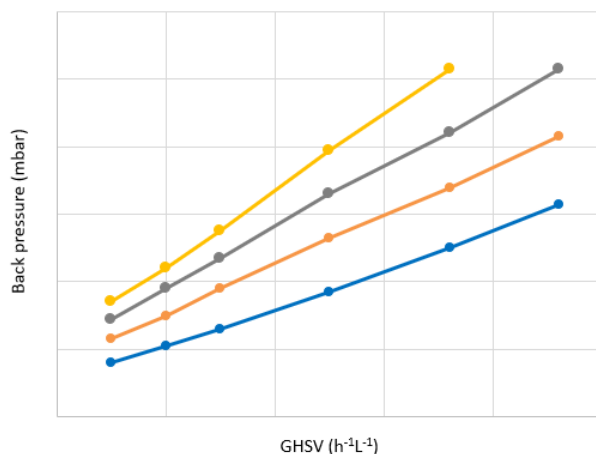


Figure 5. Back pressure was followed at different soot and ash loadings.

An example of the back pressure levels at different soot and GHSV levels for the DPF sample, in which ash had been accumulated, is shown in Figure 6.

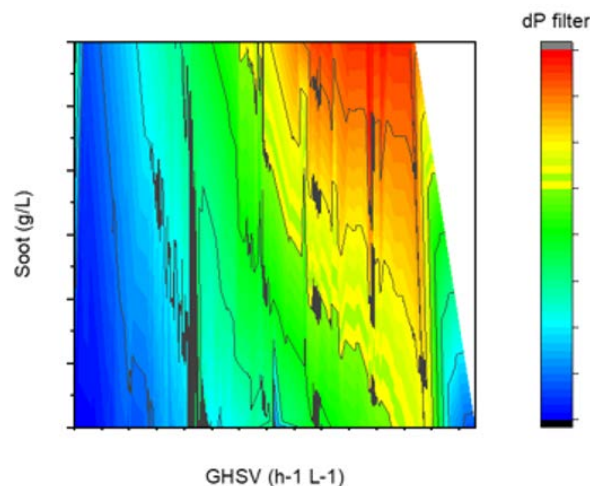


Figure 6. Back pressure was followed at different soot loadings in the GHVS tests periodically with ash accumulation in the DPF.

DPF sample was cut to inspect how smoothly ash had accumulated (Figure 7).



Figure 7. DPF sample was cut to inspect how smoothly ash had accumulated.

The soot oxidation is typically low at 200–500 °C, however, in the presence of NO₂ soot is combusted even at as low temperature as 250 °C producing CO₂ and NO. The DPF concept for high-sulphur fuels, DPF performance and its passive regeneration with NO₂ have been presented [12].

CO and soot concentrations effectively reduced in the DPF, and also NO₂ concentration originally present in engine-out exhaust reduced at temperature of 330 °C. During regeneration at 475 °C, CO, NO concentrations decreased, while NO₂ concentrations increased. This indicates that soot, CO and NO are oxidized by molecular oxygen, and NO₂ present upstream DPF also contributes in the elevated NO₂ concentrations. Soot and PN filtration efficiencies were lower during regeneration due to the conditions in clean filter. One example of the performance of clean DPF is shown in Figure 8.

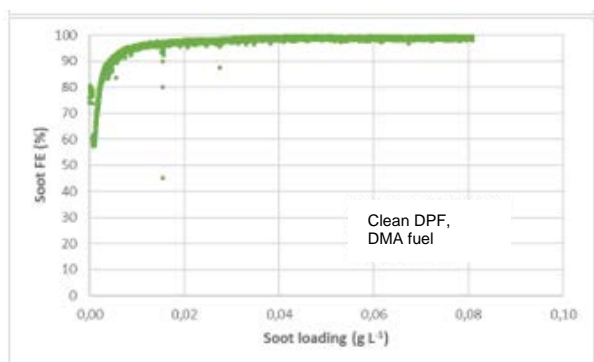


Figure 8. Filtration efficiencies of clean DPF.

4 CONCLUSIONS

Reduction of particles and black carbon emissions from marine engines is necessary to mitigate harmful effects of shipping on climate, environment and health. Particle emissions from diesel engines can be efficiently reduced with diesel particulate filter, however, its adoption to marine engines is challenging due to the poor quality of marine fuels and lubricating oils containing ash. Soot can be removed from the DPF through regeneration, but the accumulated ash is not cleaned simultaneously. Specific development of marine DPF is needed under realistic conditions using ash-containing exhaust, and the methodologies for ash generation at the laboratory scale are equally essential.

We studied three ash generating methods: 1) the burner-type ash generator was designed, built and used in the study 2) a new system for lubricating oil injection to intake air was also designed and built and 3) fuel doping with lubricating oil was the third method for ash generation. Improved method for screening of the ash concentrations in exhaust was also developed.

The highest ash concentrations in exhaust were achieved with the in-house designed and built burner-type ash generator, which tolerated high concentrations of lubricating oil in fuel. With this method, ash concentrations can be adjusted flexibly, however, the combustion is different in the burner than in diesel engine, which needs consideration. Lubricating oil injection in inlet air of a diesel engine did not produce as much ash as expected. DMB doping with high-ash lubricating oil would be suitable method to adjust ash concentration of the exhaust, however, tolerance of the high-speed engine on the doped fuel was limited. Another concept was using a robust small diesel generator, which tolerated more dopant in the DMB than the diesel engine. With the diesel generator, fuel changes were flexible and stable

soot level was achieved soon after switching to an undoped fuel.

With the three ash generation methodologies, the DPF performance tests were carried out, including sequential phases of soot accumulations, regenerations, ash accumulations and the GHSV tests. Three ash generation methods were feasible for studying how efficiently different DPFs reduced soot and particle number concentrations at different ash loadings. The methodology selected for the most extensive DPF performance study was lubricating oil doping of fuel for a robust diesel generator. Further development of ash generation methods is still needed, especially the characterisation of particulate matter, to validate its representativeness of marine diesel engine exhaust.

5 DEFINITIONS, ACRONYMS, ABBREVIATIONS

BC: Black carbon

CO₂: Carbon dioxide

DAAAC: Diesel Accelerated Aftertreatment Aging

dP: Pressure difference

DPF: Diesel particulate filter

EC: Elemental carbon

FID: Flame ionisation detector

GHSV: Gas hourly space velocity

IMO: International Maritime Organisation

OC: Organic carbon

PM: Particulate matter

SECA: Sulfur Emission Control Areas (SECA)

Sm³: Concentration at 273.15 K.

TC: Total carbon

TOA: Thermal-optical analysis

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8 CONTACT

Päivi Aakko-Saksa, VTT. E-mail paivi.aakko-saksa@vtt.fi.