

A Novel Accelerated Aging System to Study Lubricant Additive Effects on Diesel Aftertreatment System Degradation

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ABSTRACT

The challenge posed by the long run times necessary to accurately quantify ash effects on diesel aftertreatment systems has led to numerous efforts to artificially accelerate ash loading, with varying degrees of success. In this study, a heavy-duty diesel engine was outfitted with a specially designed rapid lubricant degradation and aftertreatment ash loading system. Unlike previous attempts, the proposed methodology utilizes a series of thermal reactors and combustors to simulate all three major oil consumption mechanisms, namely combustion in the power cylinder, evaporative and volatile losses, and liquid losses through the valve and turbocharger seals. In order to simulate these processes, each thermal reactor allows for the precise control of the level of lubricant additive degradation, as well as the form and quantity of degradation products introduced into the exhaust upstream of the aftertreatment system.

To demonstrate the utility of this system, individual exhaust ash and particulate samples, generated from the stock engine as well as with the accelerated ash loading system, were collected and analyzed using a number of techniques including TGA, TEM, and SEM-EDX. This analysis follows a systematic approach to understanding the similarities and differences between the ash compounds present in conventional diesel exhaust and the ash species generated using the accelerated loading system. More importantly, the results of this study provide insight into the manner in which specific accelerated oil consumption mechanisms influence particle and ash characteristics. This work presents a unique approach to the development of a realistic accelerated lubricant degradation system, in order to carefully control and condition the nature of the exhaust. Lastly, the application of this methodology to accelerate the ash loading and aging of PM and NO_x aftertreatment systems is discussed.

INTRODUCTION

The widespread use of catalyzed diesel aftertreatment systems has led to increasing concern regarding the

sensitivity of these systems to lubricant-derived ash, present in diesel exhaust. Over time, ash accumulation leads to increased flow restriction and irreversible plugging in diesel particulate filters (DPF), and various ash species are known to adversely affect the performance of diesel oxidation catalysts (DOC), catalyzed diesel particulate filters, and NO_x aftertreatment systems. While the introduction of the CJ-4 specification for diesel engine lubricants limits sulfated ash content to no more than 1.0%, the specific effects of various lubricant-derived ash-related species on aftertreatment system performance are still not well understood.

CURRENT UNDERSTANDING - In recent years, much effort has been devoted to identifying the effects of lubricant additives on ash accumulation and performance degradation of diesel aftertreatment systems (ATS). As a result, a large knowledge base has developed, leading to the following generally accepted observations:

- DPF ash increases with oil consumption and lubricant ash content [1-3].
- Lubricant-derived ash is mostly composed of oxides, sulfates, and phosphates of Zn, Ca, and Mg [1, 3-5].
- The use of lubricant sulfated ash levels to predict engine-out ash over-estimates ash emissions due to lubricant volatility [2,6,7].
- Pressure drop across the particulate filter is not indicative of total ash levels [3,7,8].
- Specific additive elements, primarily S and P adversely impact catalyst performance in addition to contributing to filter plugging [9-12].
- Ash accumulation within the DPF (plug formation versus even distribution along the channels) is affected by regeneration strategy [13,14].

Despite considerable progress, the fundamental mechanisms responsible for many of the observations listed above are, for the most part, unknown. For example, the parameters affecting ash deposition, migration, and accumulation within the channels of the DPF (plug formation, uniform distribution, and ash bridging) are little understood. Furthermore, a number of

factors are believed to affect ash packing density, which in turn impacts pressure drop; however these specific phenomena remain to be explained. Additionally, while it is apparent that lubricant additives and the composition of their resulting ash affect ash morphology, much work is still required to correlate ash characteristics to the underlying lubricant formulation. Moreover, although Ca, Mg, and Zn are generally found in similar proportions in the DPF as in the original lubricating oils, other elements such as B are not [3,15]. These few examples highlight the need for developing a fundamental understating of the specific mechanisms and parameters responsible for the widely observed, and agreed upon, ash effects listed above.

The lack of fundamental understanding is due in large part to the slow, cumulative nature of ash accumulation and its effects on ATS performance degradation. Field and laboratory tests show on the order of 100,000 miles or several thousand hours of testing are required to observe and quantify specific ash effects [7, 8]. As a result, un-accelerated testing is both costly and time consuming. While field trials provide arguably the most realistic evidence of ash accumulation and its impact on ATS performance, the inherent variability of these tests makes identifying the underlying fundamental mechanisms all the more difficult. On the other hand, laboratory tests, well-suited to conducting carefully controlled experiments, are nearly impossible to conduct given the extremely long run times required.

In response to this dilemma, researchers have developed many new and innovative test procedures for accelerating the ash exposure of diesel aftertreatment systems. A 2003 study by Lubrizol accelerated ash loading by doping oil in the fuel up to 0.2% in order to simulate 100,000 km of ash exposure over the total test duration. Four different oils were investigated, with sulfated ash levels between 2.26% and 0.63%. Similar to field-aged filters, the majority of the ash was found near the back of the filter. The use of high sulfated ash oils in the fuel doping experiments yielded ash that was denser and more evenly distributed along the filter channels as compared to lower sulfated ash oils [16]. A similar Chevron study utilized eight oils with different sulfated ash levels ranging from 1.97% to 0.43%, doped in the fuel at the 5% level. Interestingly, while Boron was found to contribute to sulfated ash in the lubricants, little Boron was found in the ash accumulated in the particulate filters [15].

In addition to fuel doping, researchers have also injected oil into the intake manifold to accelerate oil consumption and DPF ash loading. A Lubrizol study in 2004 injected oil at a rate of 1.5 ml/min. using an SAE 15W-40 1.5% sulfated ash oil. A 165 hour DPF ash loading test was conducted and the results compared to a 1,118 hour un-accelerated test. While both cases showed the ash deposited in a similar manner, the ash generated by intake oil injection appeared thicker and less porous [2]. More recent studies have utilized intake manifold oil injection with a slightly undersized DPF (DPF to engine volume ration of 0.88) to further accelerate ash loading.

These experiments increased oil consumption by 20 g/hr over the average stock engine oil consumption rate for a test duration of 120 hours. Based on these tests, the authors observed a non-linear effect of ash loading on back-pressure, with little backpressure increase until high levels of ash loading were reached [6, 17].

Still other researchers have avoided such drastic acceleration methods as fuel doping and intake manifold misting. They have instead utilized high oil consumption engine operating conditions (high speed and load) in order to somewhat accelerate DPF ash loading. This technique was employed by Exxon in a 2003 study with a 200 hr dynamometer test simulating 20,000 to 30,000 km, a 1,200 hr dynamometer test simulating 120,000 km, and a 120,000 km vehicle test for comparison [1]. A similar study was conducted by Lubrizol in 2005 in which 10 oils were evaluated, each with the same 1.8% level of sulfated ash but different additive packages. The test duration for each oil was 270 hours, simulating 27,000 km. Once again, little correlation was observed between DPF pressure drop and ash accumulation [3]. Most recently, a 2007 paper by Corning described a test procedure with relatively high engine load factors to simulate 300,000 miles and 420,000 miles over test durations of 1,720 hours and 2,640 hours, respectively. These experiments provided further evidence of the non-linear nature of DPF pressure drop as a function of both soot and ash loading [7].

While all of the studies described above utilized a wide range of techniques to accelerate DPF ash loading, differences in acceleration method, lubricant properties, test cycles, and temperature and flow histories all affect exhaust and ash characteristics and their resulting impact on aftertreatment system performance. These differences bring into question the relevance of accelerated methods to accurately simulate actual DPF ash loading. Only a small number of studies have made some comparison between the ATS ash effects observed using accelerated techniques and un-accelerated loading methods and field tests. Further, very few, if any, studies have explored the effects of acceleration method on exhaust and ash properties and applied this understanding to interpret differences in results obtained using accelerated methods and actual vehicle field tests.

Perhaps one of the more thorough investigations into the effect of acceleration method on exhaust and particle properties was conducted by Oak Ridge National Laboratory in 2005. This study compared the effects of fuel doping and intake and exhaust manifold oil injection to accelerate the phosphorous poisoning of diesel oxidation catalysts. For oil doped in the fuel or injected into the intake manifold, P, S, Zn, and O were found in small particles on the soot, whereas exhaust manifold injection coated the PM in a glaze of P, Zn, S, and O. The method of oil introduction was observed to have a significant effect on both exhaust PM composition as well as DOC poisoning [11]. Clearly, additional fundamental studies investigating the effects of acceleration

techniques on PM and ash properties and ATS impact are warranted.

In addition to gaining a better understanding of the fundamental differences between accelerated and un-accelerated ash loading methods, field trials provide valuable information and serve as benchmark for the validation of accelerated testing procedures. One of the most extensive field trials, reported in 2006, utilized 9 fuel delivery trucks retrofitted with passive DPF systems each run over 180,000 miles. This study observed differences in ash morphology and structure for vehicles using different lubricants, with lower sulfated ash oils yielding larger ash particles. Additionally, older and high mileage engines showed increased ash levels in the DPF as compared with newer engines. Similar to previous studies, exhaust backpressure was not observed to be a direct function of ash accumulation [8].

ACCELERATION METHODOLOGY AND APPROACH –

A review of the current knowledge base clearly demonstrates the need to gain a fundamental understanding of the key parameters and mechanisms responsible for the observed effects of lubricant-derived ash on diesel ATS performance. This study presents the first in a series of investigations focused on developing and validating an accelerated ash loading system through a methodical analysis of exhaust and ash characteristics, and comparison with actual engine conditions. While no accelerated ash loading method will simulate all of the effects observed in the field perfectly, well developed and validated techniques can capture the underlying fundamental processes. Furthermore, differences in PM and ash characteristics between accelerated and non-accelerated methods need to be investigated in order to better understand and quantify differences in observed ATS performance. Lastly, carefully-controlled laboratory experiments are required if progress related to uncovering the underlying fundamental mechanisms influencing ash effects on ATS is to be made.

In order to realistically accelerate lubricant-derived ash loading of diesel aftertreatment systems, it is instructive to understand the major oil consumption mechanisms in a heavy-duty diesel engine:

1. Transport through the piston ring pack leading to combusted and volatile losses.
2. Valve and turbocharger seal leakage leading to liquid oil losses.
3. Blowby and loss through PCV system, if used [18].

The approach taken in this work focuses on three key aspects. First, the effects of specific acceleration methods on PM and ash characteristics are investigated, so that differences between accelerated and non-accelerated techniques can be accounted for. Second, investigations into the effects of specific oil consumption mechanism (combusted lubricant, and volatile and liquid losses) on engine-out PM and ash characteristics are required in order to more accurately accelerate lubricant

consumption. As the combustion of lubricant in the power cylinder is the single largest source of diesel engine oil consumption, this paper focuses on how lubricant combustion affects ash properties. Lastly, ATS performance and ash characteristics evaluated using the accelerated methodology must be compared with field aged units in order to understand and minimize any significant differences.

EXPERIMENTAL DESCRIPTION

The experiments described in this study were conducted utilizing fuels and lubricants of known composition. Bulk and individual particulate and ash emissions were sampled from the exhaust stream and extensively characterized using a number of advanced techniques.

ENGINE AND INSTRUMENTATION - The engine used in this study was a pre-production Cummins development engine based on the 2002 ISB 300 engine platform. The ISB 300 is a 6 cylinder, 5.9 liter, turbocharged, direct injection diesel engine. The engine is rated at 224 kW (300 hp) at 2500 rpm and 890 N-m (660 ft-lb) at 1600 rpm. In order to meet 2002 EPA emissions standards, the engine is equipped with a high-pressure common rail fuel injection system, variable geometry turbocharger, and cooled exhaust gas recirculation (EGR). Table 1 contains additional details regarding engine specifications and geometry.

Table 1. Cummins ISB 300 engine specifications.

Model	ISB 300
Maximum Torque	890 N-m @ 1600 rpm
Maximum Power	224 kW @ 2500 rpm
Number of Cylinders	6, in-line
Combustion System	4 stroke, direct injection
Injection System	Common Rail
Aspiration	Variable geometry turbocharger and intercooler
Displaced Volume	5.9 liters
Compression Ratio	17.2:1
Cylinder Head Layout	4 valves/cylinder
Injection Nozzle	O.D. = 158 µm
	L = 1.00 mm
	8 sac-less nozzles per injector
Injection Pressure	800-1600 bar

ACCELERATED ASH LOADING SYSTEM – The accelerated ash loading and aftertreatment aging system was designed to load two 0.3L DPF test sections to 40 g/L of ash in 10 hours, in parallel. Using a CJ-4, 1.0% sulfated ash oil requires an oil consumption rate of approximately 340 g/hr. Ash levels of 40 g/L have been reported to correspond to roughly 300,000 miles of on-road use [7]. In addition to accelerating DPF ash loading, this system is also designed to accommodate various DOC and NOx aftertreatment systems. The accelerated ash loading system is shown in Figure 1.

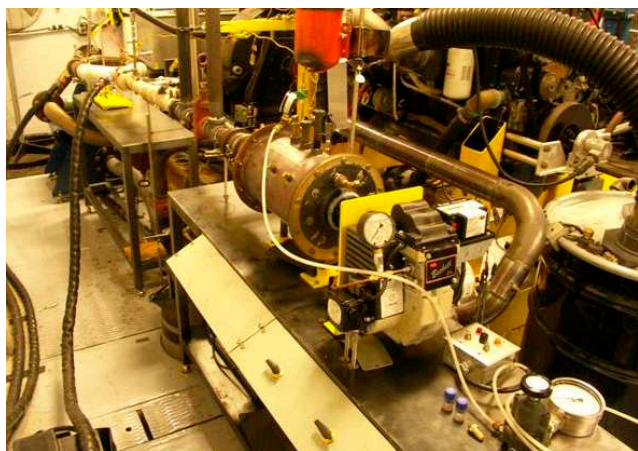


Figure 1. Experimental set-up showing accelerated ash loading system mounted beside Cummins ISB test bed.

The heart of the system is composed of an industrial diesel burner and combustion chamber. Lubricant is introduced into the combustion chamber by doping in the burner's fuel or via injection using a separate air assisted injector. Volatile lubricant products are created in a separate thermal reactor and can also be supplied to the combustion chamber at a known flow rate. A second air-assisted oil injector mounted downstream from the combustion chamber enables the introduction of a small fraction of liquid oil into the exhaust. This system, therefore, allows for the simulation and control of all three major oil consumption mechanisms, namely combustion in the power cylinder, and liquid and volatile losses. Table 2 presents the system's operating specifications and Figure A-1 in the Appendix depicts the lay-out and provides additional details of this system.

Table 2. Accelerated ash loading system specifications.

Fuel Consumption	1.5 - 7.6 l/hr
Injection Pressure	6.9 - 13.8 bar
Oil Consumption	0.94 - 9.4 ml/min
Air Flow	266 - 1130 SLPM
ATS Inlet Temperature	200 - 800 °C

A heat exchanger mounted downstream of the combustor provides additional control over exhaust temperature, independent of the burner settings, allowing for online DPF regeneration. A junction between the combustor outlet and heat exchanger inlet connects the Cummins exhaust system to the accelerated ash loading rig. A portion of the ISB's exhaust is routed to the ash loading system, providing a realistic exhaust composition (gaseous and PM) that is mixed with the ash and PM generated by the combustor. Following the heat exchanger, the mixture of engine exhaust and ash is fed into the aftertreatment test section.

The purpose of the accelerated ash loading system shown in Figure 1 is only to supplement the Cummins exhaust with elevated ash levels. Careful control of the various accelerated oil consumption mechanisms enables the simulation of engine-out ash and PM as closely as possible. The objective of this work was to

investigate the effect of oil introduction method on ash and PM properties entering the aftertreatment system. Critical to accurate simulation and acceleration of ash emissions is an understanding of how each specific accelerated oil consumption mechanism affects ash properties, and, more importantly, how this synthetic ash compares to the ash generated by the test engine alone.

PM SAMPLING AND CHARACTERIZATION – PM samples were collected from the Cummins ISB as well as the accelerated ash loading system. All particulate samples were collected from the raw exhaust upstream of the aftertreatment system. This allowed for the characterization of the particles and ash as they exist in the raw undiluted exhaust entering the particulate filter. Bulk PM samples were collected on 47 mm glass fiber filters and particulate emission rates were determined gravimetrically. Following gravimetric analysis, the composition of the particles was characterized using Horiba's MEXA 1370 PM analyzer. The MEXA 1370 measures PM sulfate fraction (SO₄), soluble organic fraction (SOF), and insoluble fraction (SOL).

In addition to bulk PM sampling and analysis, individual particulate samples were also collected from the raw exhaust. The particles were collected directly on 3 mm (0.12 inch) diameter grids, fixed to a sample probe and inserted through a sample port on the exhaust system upstream of the DPF. The use of the small sampling grids allowed for the collection of individual particles frozen in composition and structure, as they exist in the exhaust stream entering the ATS. Further, this collection method serves as an ideal means for subsequent analysis of PM composition and morphology via electron microscopy. A similar system has been developed and used at Argonne National Laboratories applied to the study of PM morphology, with additional details available in [19-22].

Transmission Electron Microscopy – Transmission electron microscopy (TEM) provides high resolution images of PM samples to a fraction of a nanometer. The use of commercial image processing software enabled the determination of average primary particle and agglomerate sizes and additional morphological information. In contrast to commercial particle size measurement systems based on aerodynamic or mobility diameter measurements (which assume spherical, agglomerates), TEM allows for the measurement of the true geometric dimensions of primary and agglomerate particles [21]. Previous studies have shown large variations in particle geometries from long chain-like structures to more spherical clusters. Further, agglomerate sizes and geometries have also been shown to vary with engine operating condition, engine type, and location along the exhaust system, among other factors [19-22].

PM Size Measurement – The PM size measurements from the images obtained via TEM were processed using commercially available image processing software. Two dimensions were measured for each agglomerate and

primary particle as shown in Figure 2. Previous work measuring primary particle size at two different viewing angles of 0° and 50° showed primary particles to be nearly spherical [21]. On the other hand, agglomerate particles, as shown in Figure 2, generally do not fit a simple geometric description.

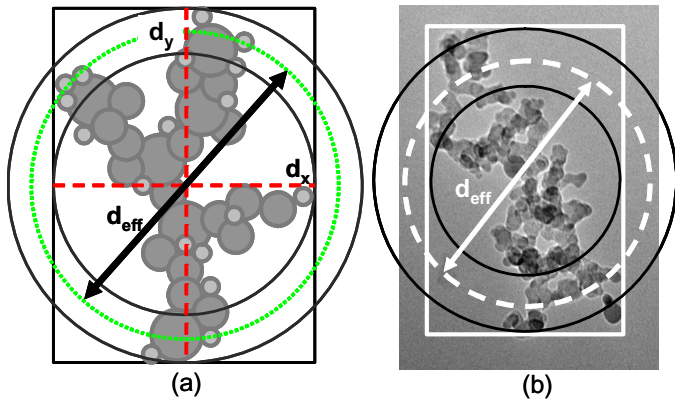


Figure 2. Measurement methodology for determination of agglomerate effective diameter (a) conceptual representation and (b) PM agglomerate at 27,000X.

The two dimensions measured for each agglomerate, d_y and d_x , were used to compute an effective diameter, d_{eff} , as follows:

$$d_{eff} = \frac{d_x + d_y}{2} \quad (1)$$

Additionally, the geometry of the agglomerate can be described by the following modified aspect ratio:

$$1 \leq \frac{\max(d_x, d_y)}{d_{eff}} < 2 \quad (2)$$

where perfectly spherical particles exhibit a ratio of 1 and long chain-like agglomerates approach 2. While measurements of radii of gyration and fractal geometries are often used to describe agglomerate dimensions in the literature, the limitations of the image processing software used in this study required the use of the simple measurements described above. Nonetheless, the measurements appear adequate for the comparative purposes of this work.

ASH SAMPLING AND CHARACTERIZATION – Ash samples from the accelerated loading system were characterized and compared with ash collected from the Cummins ISB as well as ash removed from field-aged DPFs.

Thermo-Gravimetric Analysis - Thermo-gravimetric analysis (TGA) was used to measure the total ash content in the PM samples, without regard to specific ash composition. TGA measures the change in weight of a sample as a function of temperature. The ash content of a particulate sample is determined as the amount of incombustible material remaining following controlled oxidation. The Perkin Elmer TGA 7 used in this study has a maximum temperature of 1000 °C with a sensitivity

of $\pm 1 \mu\text{g}$. Additional details regarding the measurement of ash content in PM and the specific method used in this study are described in [3]. The ash generated from the PM samples via TGA was also subjected to microscopy analysis.

Scanning Electron Microscopy - Scanning electron microscopy (SEM) was used to characterize ash morphology and composition for ash in the exhaust entering the DPF, as well as ash collected from field-aged filters. In contrast to TEM, SEM provides high resolution, three-dimensional images of the ash. In addition to imaging, energy dispersive x-ray analysis (EDX) used in conjunction with the SEM allowed for the identification of specific elements. This technique, thus, provided qualitative information on ash composition and distribution of lubricant elements in the ash samples.

FUEL AND LUBRICANT PROPERTIES – The fuels and lubricants were subject to a detailed elemental analysis in order to accurately quantify lubricant speciated emission rates and to account for the presence of any trace elements in the fuel. A SAE 15W-40 commercially available CJ-4 diesel engine oil was used in both the Cummins ISB as well as the accelerated ash loading system. The elemental composition of the lubricant is listed in Table 3.

Table 3. Lubricant elemental composition.

ASTM D5185							
Lubricant	B	Ca	Fe	Mg	P	Zn	S
CJ-4 [ppm]	586	1388	2	355	985	1226	4606

Both the Cummins ISB and the diesel burner on the accelerated ash loading system were fuelled with an ultra-low sulfur diesel (ULSD, 15 ppm S). The elemental composition of the fuel is shown in Table 4 below.

Table 4. Fuel elemental composition. All values below minimum detectable limits.

ASTM D5185						
Element	Ca	Mg	P	Na	K	Zn
ULSD [ppb]	<97	<56	<1180	<2010	<2690	<155

TEST MATRIX AND PROCEDURES – The ash emissions from the Cummins ISB were extensively characterized in a previous study [23] and thus provided a good baseline for comparison with ash generated from the accelerated loading system. Additionally, CJ-4 lubricant additive elemental emission rates were also measured for this engine, using neutron activation analysis, and the results reported in [24].

In order to specifically identify the effects of accelerated lubricant consumption via combustion method on ash and PM characteristics, the accelerated loading system was used without the addition of supplemental exhaust from the Cummins ISB. Further, only liquid oil was introduced into the combustor by one of two methods: fuel doping, and direct injection.