

Lubricant-Derived Ash Impact on Gasoline Particulate Filter Performance

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ABSTRACT

The increasing use of gasoline direct injection (GDI) engines coupled with the implementation of new particulate matter (PM) and particle number (PN) emissions regulations requires new emissions control strategies. Gasoline particulate filters (GPFs) present one approach to reduce particle emissions. Although primarily composed of combustible material which may be removed through oxidation, particle also contains incombustible components or ash. Over the service life of the filter the accumulation of ash causes an increase in exhaust backpressure, and limits the useful life of the GPF. This study utilized an accelerated aging system to generate elevated ash levels by injecting lubricant oil with the gasoline fuel into a burner system. GPFs were aged to a series of levels representing filter life up to 150,000 miles (240,000 km). The impact of ash on the filter pressure drop and on its sensitivity to soot accumulation was investigated at specific ash levels. Ash deposits and interactions with the filter substrate were studied using x-ray micro computed tomography (CT), focused ion beam milling (FIB), cross section ion milling (CSIM), scanning electron microscope imaging (SEM), and energy dispersive x-rays (EDX). This extensive post-mortem dataset was used to quantify the extent of the substrate-ash interactions. The results highlight the importance of understanding ash impacts on the backpressure and soot loading of GPFs over their full useful life.

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INTRODUCTION

The transportation sector consumes 28% of total energy usage in the United States and contributes 28% of US greenhouse gas (GHG) emissions [1]. Vehicles powered by gasoline internal combustion engines consume 57% of U.S. transportation energy [2]. Reducing GHG emissions has become an important part of curbing climate change. In April 2010, the U.S. Environmental Protection Agency (EPA) and National Highway Traffic Safety Administration (NHTSA) established final rules for phase one on the Light-Duty Vehicle Greenhouse Gas Emissions and Corporate Average Fuel Economy Standards (CAFE) setting goals for reduction of CO₂ emissions and increase in fleet average fuel economy [3]. The final rule for phase two of this effort was passed in October 2012. CAFE standards aim to reduce the fleet average CO₂ emissions for light-duty vehicles by 45% between model year (MY) 2012 and 2025 with an associated fleet average fuel economy increase of 81% [3, 4].

To address vehicle emissions other than GHG, the EPA will implement Tier 3 Motor Vehicle and Fuel Standards in 2017. This regulation will set strict limits on vehicle tailpipe emissions of non-methane organic gasses (NMOG), nitrogen oxides (NO_x), and particulate matter (PM). Tier 3 standards aim to reduce NMOG+NO_x emissions levels by 70% and PM emissions by 80%, relative to current fleet averages. These standards will be phased in from MY2017 to MY2025 concurrent with the phase in of CAFE standard [5]. For Tier 3 regulations all PM limits are stated in terms of total mass of particle emissions.

For the European market, Euro 6 emissions standards were adopted in 2007. Euro 6 includes PM limits by mass for gasoline direct injection (GDI) engines that are of the same order of magnitude as U.S. Tier 3 standards. However, Euro 6 includes an additional limitation on particle number (PN) emissions of 6.0×10^{11} particles/km. This requirement will phase in for GDI engines with an initial standard of 6.0×10^{12} particles/km taking force in 2015 and the full

Euro 6b limit of 6.0×10^{11} particles/km beginning in 2018 [6]. Analysis based on both average particle mass as well as emissions levels for current GDI engine technology suggest that the Euro 6c PN limit requires the largest particulate emission reduction from current levels [7, 8].

Concurrent phase-in of these emissions regulations has created a new challenge for gasoline spark ignition engines installed in light-duty vehicles. CAFE standards motivate increased use of gasoline direct injection fuel systems for increased fuel efficiency. However, GDI engines can emit higher levels of PM than port injection engines currently in widespread use [9]. PM emissions levels from GDI engines can be at or above the PM limits imposed in Tier 3 and Euro 6 emissions standards requiring new PM mitigation strategies for GDI engines in order to realize the potential fuel economy improvements while maintaining regulatory compliance.

Gasoline Particulate Filters (GPF) are one PM mitigation strategy that can be used to reduce PM and PN emissions levels to meet Tier 3 and Euro 6 standards [10]. Because engine design and operation strategies may successfully reduce emissions levels sufficiently to meet U.S. and European standards, it is important to understand the impact of GPF implementation over the course of the vehicle's service life [10,11,12].

GPF systems introduce added exhaust system backpressure that reduce power output and fuel economy, which in turn increase CO_2 emissions [12]. The backpressure generated in a GPF system increases during the filter service life due to deposition of inorganic ash particles derived from incombustible oil additives, engine wear particles, and other secondary sources. This study aims to understand the impact of ash deposition in GPFs using an accelerated aging system to accumulate oil-derived ash in GPF samples, simulating extended on-vehicle operation under carefully-controlled conditions, for testing and analysis.

ACCELERATED GPF AGING

Application of conventional particulate filter technology, previously developed for diesel applications, to GDI engines requires examination of key differences between diesel and gasoline particle and ash emissions in order to understand GPF performance across the entire useful life of the system. While GPFs can be installed on test vehicles for aging, this approach has several drawbacks. First, long run times are required to complete 150,000 road miles (240,000 km) of operation to reach the intended service life limit of the GPF. Second, the exhaust conditions that the GPF is exposed to cannot be controlled and varied in a systematic fashion on the vehicle over an extended test. An accelerated aging system was, therefore, developed to reduce the time required to age GPF samples and provide for control and monitoring of operating temperatures, pressures, and flow rate during the aging process [8].

A diagram of the accelerated aging system is included in [appendix A, Figure A1](#). The system utilizes a gasoline powered continuously spark ignited burner system to generate exhaust gases. Engine oil is introduced into the gasoline burner combustion chamber in an atomizing spray to generate elevated levels of oil-derived ash

constituents in the exhaust stream. A water-cooled heat exchanger with temperature control valve controls the temperature at the GPF inlet. Flow through the system is driven by a centrifugal blower located downstream of the GPF outlet. The accelerated aging system is instrumented to measure airflow rate, filter inlet and outlet static pressure, and inlet and outlet gas flow temperature.

Filter Loading Test Matrix

Five identical cordierite wall flow monolith GPFs were loaded with ash deposits using the accelerated aging system to characterize the pressure drop performance over the filter service life. Filters were loaded to terminal ash loads for 15,000 to 150,000 simulated road miles (24,000 - 240,000 km) of operation. The filter loaded to 150,000 road miles was also loaded with soot from a Ford 1.6 L EcoBoost engine at regular intervals during its aging. The fundamental characteristics of the filters tested are shown in [Table 1](#).

Table 1. Test Filter Characteristics

Filter Parameters	
Material	Cordierite
Length (in)	5.51
Diameter (in)	4.66
Channel Density (CPSI)	220
Volume (L)	1.54
Catalyst Coating	None
Porosity	48%
Mean Pore Size (μm)	12
Wall Thickness (μm)	150

The fundamental measure of filter age in this study is grams of ash present in the filter per liter of filter volume. The objective of the loading experiment is to generate filter samples with terminal ash loading levels equivalent to 15,000, 30,000, 60,000, 100,000, and 150,000 road miles. The specific ash levels in the GPFs were verified gravimetrically in this study. The methodology to estimate the equivalent road miles for the filters aged on the accelerated loading system is given by [Equation 1](#).

$$AL = \frac{OilCons * \rho_{oil} * \tilde{x}_{ash}}{V_{filter}} * FR_{ash} * Mi_{sim} \quad (1)$$

AL - Target Ash Load (g/L)

$OilCons$ - Engine Oil Consumption (L/km)

ρ_{oil} - Oil Density (g/m^3)

$\tilde{x}_{ash}$ - Mass Fraction Incombustible Ash in Test Oil

V_{filter} - Filter Volume (L)

FR_{ash} - Ash Finding Rate

Mi_{sim} - Simulated Distance (mi)

The first term of equation 1 determines the theoretical rate of oil-derived ash in the exhaust stream based on engine and oil characteristics and normalized to the filter volume. Research has shown that this theoretical rate of ash emissions from the engine overestimates the rate at which ash is found to deposit in particulate filters in experimental tests. The ratio of the actual ash loading rate to the theoretical ash emission rate is known as the ash finding rate. Finding rates ranging from 5-65% have been reported in previous studies [13,14,15]. The assumed finding rate for this experiment was calculated based on the concentration of individual oil additives in the test oil, determined through ASTM D874, and finding rates for individual additives reported in [13]. The reduced ash finding rate is attributed to differences in volatilities and emission rates of various additives, which is not necessarily equivalent to the initial additive concentrations in the engine sump. Table 2 summarizes the values used in equation 1 and their sources.

Table 2. Target Ash Load Calculation; Assumed Values

Input	Value	Source
Oil Consumption	0.00016 (L/Km)	Engine OEM
Oil Density	877 (kg/m ³)	Measured
Mass Fraction Ash	0.0102	ASTM D874
Filter Volume	1.54 (L)	Measured
Ash Finding Rate	37%	[13]

The target ash loads for five test filters were calculated using equation 1 and are given in Table 3.

Table 3. Target Ash Load for Five Test Filters

Filter	Final Load (g/L)	Equivalent Miles
1	3	15,000
2	6.1	30,000
3	12.2	60,000
4	20.3	100,000
5	30.5	150,000

Ash Loading Cycle

There are two possible GPF installation locations in the vehicle exhaust system. The close-coupled position locates the GPF directly downstream of the three-way catalyst (TWC). This places the GPF in the engine compartment with only the TWC between it and the engine exhaust manifold. The underbody position places the GPF in-line with the exhaust piping beneath the vehicle chassis upstream of the muffler. These positions offer various advantages and disadvantages for ease of installation and access, but the primary impact on filter performance is the difference in exhaust gas temperature when it reaches the GPF. The exhaust stream will be significantly cooler when it reaches an underbody mounted GPF than when it exits the TWC and enters a close-coupled GPF.

This loading experiment, described in this work, simulates an underbody GPF position. The GPF is exposed to a maximum temperature of 650°C and the average loading temperature is 400°C. The loading cycle utilizes a periodic regeneration strategy. The test cycle is summarized in Table 4.

Table 4. Test Cycle Parameters

Primary Loading Period	3 hr 30 min
Loading Temp (°C)	400
Regeneration Period	30 min
Regeneration Temp (°C)	600-650
Flow Rate (SCFM)	32-35
Space Velocity (1/hr)	35-38K
Ash Loading Rate (g/L-hr)	0.25

Soot (produced by the gasoline burner) accumulated during the primary loading phase of the cycle was oxidized by elevating the filter inlet temperature and maintaining high (5%) oxygen content in the exhaust stream during the regeneration phase. At the conclusion of each loading cycle the filter was weighed hot, at 170°C, to determine the mass of ash added during the cycle. After weighing and cooling the filter, the pressure drop was measured on a cold flow test bench over a range of flow rates representative of actual on-engine operation. In some cases, two loading cycles were completed in series prior to weighing the filter and measuring pressure drop.

Pressure Drop Measurements

The pressure drop across the filter was measured after each ash loading cycle or series of two loading cycles. The number of cycles between pressure drop measurements determines the resolution of pressure drop data with respect to ash load.

Pressure drop measurements were always taken with the filter and airflow at room temperature, which ranged from 20-35°C. Flow through the filter is driven entirely by the downstream centrifugal blower. The burner system and water-cooled heat exchanger are bypassed and isolated for all pressure drop measurements. The flow rate through the filter is varied from 0-80 SCFM (0-2265 SLPM) in 5-10 SCFM (141-282 SLPM) steps by varying the blower speed. The airflow rate is measured at the system inlet and static pressures are measured immediately upstream and downstream of the GPF housing. Pressure drop across the filter is calculated and the resulting data is fit with a quadratic function to model pressure drop versus flow rate for the given filter ash loading level. Figure 1 shows raw data and quadratic fit lines for each pressure drop measurement for filter 1.

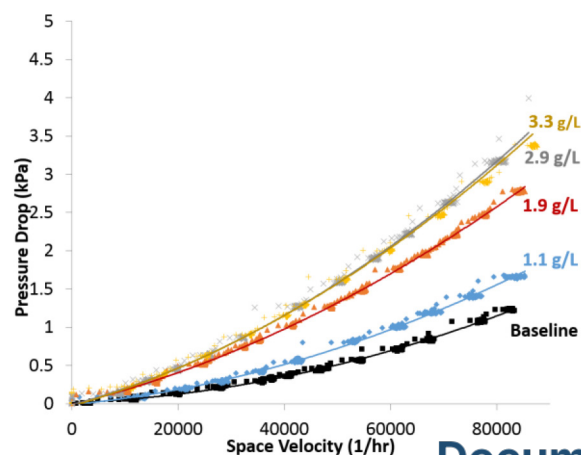


Figure 1. Filter 1; Complete plot of pressure drop vs. flow rate raw data and quadratic fit model for varying filter ash levels.

The gravimetric ash loading levels corresponding to each pressure drop measurement are also listed in [Figure 1](#). The results show a high degree of sensitivity to relatively low (< 3 g/L) levels of ash accumulation in the filter.

Soot Loading Cycle

Filter 5 (see [Table 3](#)) was loaded to a terminal ash load of 30.2 g/L simulating 150,000 road miles (240,000 km) of operation. The filter was also loaded with soot from a 1.6 L Ford EcoBoost engine at regular intervals during the ash loading process. Soot loading for filter 5 was conducted in order to characterize changes in the filter's pressure drop response to soot loading across its service life.

A soot loading cycle consisted of loading the GPF from 0-3 g/L of soot in 0.5 g/L intervals and measuring the resulting pressure drop for each loading interval. A soot loading cycle was conducted on the clean filter prior to ash loading on the accelerated loading system. During aging a soot loading cycle was completed at 5 g/L ash load intervals. Pressure drop and weight measurements to determine soot mass additions were conducted following the same procedures used for ash loading.

At the conclusion of a soot loading cycle, the soot in the filter was regenerated using the accelerated aging system burner with no oil injection. The filter was reweighed after regeneration and an additional pressure drop measurement was completed using the cold flow bench. For each soot loading cycle a small mass increase was observed from the initial zero soot mass prior to soot loading to the final zero soot mass after regeneration. This mass increase was treated as additional ash in the filter.

INVESTIGATIONS OF ASH IMPACTS

Ash-Substrate Interactions

The GPFs which were ash loaded in this study were investigated with a number of non-destructive and destructive tools in order to understand the nature of ash-substrate interactions and associated influences on filter pressure drop. General imaging and elemental analysis were performed by an FEI/Philips XL30 Field Emission Gun (FEG) Environmental Scanning Electron Microscope (ESEM) with an EDAX Energy Dispersive X-ray Spectroscopy (EDX) detector. A conductive coating on SEM samples was not required with the use of the ESEM and detailed use of this instrument was discussed in [\[16\]](#). Three dimensional data were collected with an X-tek HMXST 225 microCT X-ray Computed Tomography instrument, which was discussed in detail in [\[17\]](#). Cross sections of the GPF samples were prepared on the mm-scale by a JEOL Cross Section Polisher (CSIM) which uses argon ions and on the μm -scale by a Helios Nanolab 600 Dual Beam Focused Ion Beam Milling System (FIB) which uses gallium ions and was discussed in detail in [\[18\]](#).

Post mortem investigations of the GPFs were done on several relative size scales due to the fact that relevant ash-substrate interactions occur over a wide size range, from sub- μm to tens of cm. Filters were first scanned via X-ray CT to produce three dimensional representations of the ash accumulation within the filters, and to

provide accurate statistical measurements for the ash plug lengths. The filters were then cut into quarters and small, less than 1 cm^3 , samples from the middle and rear (axial direction) were removed from one of the quarters for destructive analysis and imaging. The ESEM was used in backscattered electron detector mode in order to increase the contrast between the ash and filter substrate, in addition to the EDX detector to confirm the locations of ash. The ESEM was used for imaging of both inlet channel surfaces and wall cross sections prepared by the CSIM. Like the FIB, the CSIM fires ions at the sample surface to induce controlled sputtering of the sample, however the CSIM uses less powerful argon ions while the FIB uses gallium ions. Cross sections were prepared by both the CSIM and FIB in order to directly observe ash penetration into the filter wall and into the filter surface pores, respectively.

PRESSURE DROP RESULTS

Ash-Loaded Filters

Using the quadratic pressure drop models generated from the raw pressure drop data (similar to [Fig. 1](#)), the expected pressure drop can be calculated for any flow rate between 0 and 80 SCFM (0-2265 SLPM). For a given flow rate or equivalent space velocity, the pressure drop is plotted against ash load to represent the increase in pressure drop over the service life of the filter. [Figure 2](#) presents a compilation of pressure drop versus ash load for filters 1 through 5. All filters were fully regenerated to remove soot prior to these measurements.

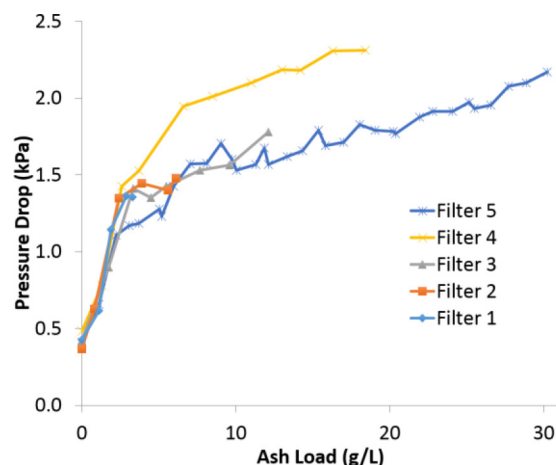


Figure 2. Filters 1-5; Pressure drop vs. ash load at 45,000 hr⁻¹.

All filters follow the same steep slope during the first 4 g/L of ash loading. This steep rise in filter pressure drop is associated with the deep bed filtration regime during which ash particles penetrate into the filter wall reducing overall porosity and restricting the pores. For ash loads greater than 8 g/L filters 4 and 5 display similar slopes, 0.032 and 0.025 kPa per g/L respectively. This area of reduced slope and linear pressure drop increase represents the cake layer filtration regime. In this regime ash builds up in a layer on the surface of the filter walls causing an increase in pressure drop proportional to the thickness in the ash deposit layer.