Integrated Selective Catalytic Reduction−Diesel Particulate Filter Aftertreatment: Insights into Pressure Drop, NO$_x$ Conversion, and Passive Soot Oxidation Behavior

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ABSTRACT: Integrating urea-selective catalytic reduction (SCR) and diesel particulate filter (DPF) technologies into a single device has the potential to reduce the complexity of current diesel aftertreatment strategies. Fundamental studies were performed to shed light on the pressure drop and reaction behavior of integrated SCR and DPF systems. Details of SCR washcoat amount and location were investigated for effect on pressure drop during soot filtration. The SCR catalyst primarily impacted depth filtration of soot, promoted by increased catalyst located within the upstream portion of the porous filter wall. This effect is believed to be related to the nature of the porous filter substrate and pore network and changing of the rate at which pores plug in the presence of catalyst. SCR catalyst on the wall of the inlet filter channel also had an effect on the pressure rise during cake filtration of soot. NO$_x$ reduction efficiency measurements were performed to determine the nature and magnitude of the effect of soot on SCR performance. The effect of soot on the SCR performance is primarily attributed to the contribution of passive soot oxidation, and the propensity for soot oxidation to shift the NO$_2$/NO$_x$ fraction relative to 0.5. SCR performance at NO$_2$/NO$_x <$ 0.5 is adversely affected by the presence of soot oxidation by increasing the SCR dependency on standard (NO only) SCR reactions; conversely, at NO$_2$/NO$_x >$ 0.5, the SCR performance is positively impacted by a decreased dependency on NO$_2$-only SCR reactions. Temperature-programmed oxidation studies were performed to evaluate the impact of SCR on passive soot oxidation. SCR adversely impacts soot oxidation performance via NO$_2$ diffusive effects, decreasing NO$_2$ concentration in the inlet channel. This impact can be minimized or recovered at higher NO$_2$ concentration and NO$_2$/NO$_x$ fractions >0.5.

1. INTRODUCTION

The diesel engine is currently the primary engine employed in the long- and short-haul commercial trucking industry because of its attractive wear characteristics, increased engine durability, and ability to deliver power efficiently under high load conditions. Manufacturers are planning much more widespread usage than historically employed, motivated by reduced fossil fuel consumption, reduced CO$_2$ emissions, and the ability to operate on biologically derived fuels. However, for such a paradigm shift to take place, there are hurdles to overcome. Diesel close-coupled injection, compression, and ignition result in a heterogeneous two-phase burn that takes place largely at fuel droplet edges where the fuel and the air are mixing. The result of this is a near-stoichiometric burn that is very hot and comparatively long in duration, leading to elevated levels of NO$_x$ and PM formation. Engine controls and advanced fuel injection and combustion strategies have made significant strides in lowering diesel engine emission levels. However, persistently high particulate matter (PM) and nitrogen oxides (NO$_x$) emissions dictate that exhaust aftertreatment will be a necessity.

Exhaust aftertreatment reduces pollutants from the engine exhaust by treating the exhaust gas in a thermally and chemically controlled environment. Widespread introduction of advanced diesel exhaust aftertreatment is necessary for simultaneous compliance with NO$_x$ and particulate matter emission standards. Aftertreatment may lessen the trade-offs involved in controlling both NO$_x$ and PM emissions while minimizing losses in fuel efficiency.

Several existing emission control technologies have proven effective at controlling emissions individually. A diesel oxidation catalyst (DOC) effectively oxidizes NO, CO, and hydrocarbon (HC) emissions, and the diesel particulate filter effectively removes particulate matter from the exhaust stream. The wall-flow diesel particulate filter is a honeycomb monolithic structure made from porous ceramic that has channels alternately plugged at the ends so that exhaust gas is forced through the porous channel wall. As engine exhaust is passed through a DPF, particulates are retained on the upstream portion of the filter wall and accumulate over time. Soot filtration first occurs by depth filtration, which is the collection of soot within the porous microstructure of the filter wall. This is typically accompanied by a sharp increase in the pressure drop across the filter. However, filtration will usually quickly transition to cake filtration, which is when soot is no longer accumulating within the wall microstructure, but rather, on the upstream surface of the filter wall. In this instance, the soot cake present on the upstream filter wall serves as the filter, and this is typically accompanied by a smaller increase in pressure drop across the filter. Together, these lead to an increase in pressure drop across DPF and, thus, an increase in the back pressure on the engine that adversely affects the engine operation and fuel consumption.

Selective catalytic reduction (SCR) has been developed as an effective technology at reducing NO_x emissions under lean burn conditions. SCR catalysts selectively reduce NO_x to N_2 and H_2O through the use of NH_3 as the reductant. NH_3 is supplied via a urea injection system and subsequent thermal decomposition to isocyanic acid and 1 equiv of NH_3, as shown in eq 1. A second equivalent of NH_3 is produced from the surface-catalyzed decomposition of isocyanic acid, as shown in eq 2.

$$NH_2-CO-NH_2 \rightarrow NH_3 + HNCO \qquad (1)$$

$$HNCO + H_2O \rightarrow NH_3 + CO_2 \qquad (2)$$
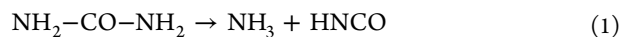
The SCR process begins with reversible ammonia chemisorption on catalytic Brønsted acid sites, where it becomes available for SCR reaction. The main NH_3−SCR reactions promoted by Fe- and Cu-exchanged zeolites are shown in eqs 3−5 and consist of the standard SCR reaction (NO only), the fast SCR reaction (equimolar NO and NO_2), and the NO_2-only SCR reaction, respectively. Zeolite-based catalysts (such as vanadium systems) exhibit maximum NO_x reduction activity at $NO_2/NO_x = 0.5$ as a result of the prevalence of the fast SCR reaction. The standard SCR reaction participates at $NO_2/NO_x < 0.5$ because of the relative abundance of NO; similarly, the NO_2-only SCR reaction participates at $NO_2/NO_x > 0.5$ because of the relative abundance of NO_2.

$$4NH_3 + 4NO + O_2 \rightarrow 4N_2 + 6H_2O \qquad (3)$$

$$2NH_3 + NO + NO_2 \rightarrow 2N_2 + 3H_2O \qquad (4)$$

$$4NH_3 + 3NO_2 \rightarrow 3.5N_2 + 6H_2O \qquad (5)$$
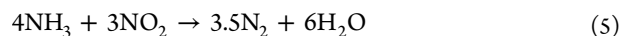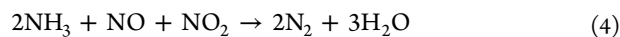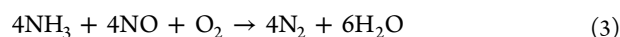
Current focus in the U.S. has been on development of Fe- and Cu-based zeolite formulations as effective SCR catalysts for meeting NO_x emission regulations.[1,2] Early zeolite catalysts favored Fe-exchanged versions (Fe−ZSM-5) with medium-sized pores (~5.5 Å) for heavy-duty diesel (HDD) applications for their improved activity above 350 °C and superior thermal stability as compared with Cu-exchanged versions, which had previously been confined to light-duty diesel (LDD) applications. Fe zeolites exhibit less sensitivity to $NO_2/NO_x >$ 0.5 because of the efficiency of the NO_2-only SCR reaction on these catalysts;[3,4] however, recent advancements in Cu-exchanged small pore zeolites have made them more much attractive for both HDD and LDD applications.[5] Specifically, the small pore Cu-exchanged chabazite-type zeolites Cu-SSZ-13 and Cu-SAPO-34 have recently exhibited good activity over a broad temperature range, good thermal stability, and good resistance to hydrocarbon deactivation.[6−10] These Cu zeolites are shown to exhibit less sensitivity to $NO_2/NO_x < 0.5$[11] and have also demonstrated improved SCR selectivity to N_2 versus undesirable products, including N_2O.[7]

Integration of catalytic SCR and DPF technologies is a relatively new area in exhaust aftertreatment systems. Integrated SCR/DPF technology combines NO_x reduction and soot filtration in a single 2-way device. Its development is motivated by emission compliance in a manner that reduces aftertreatment system volume and cost and increases packaging flexibility. As such, it is currently being pursued for both LDD and HDD diesel applications.

Figure 1 shows a comparison of wall-flow and flow-through substrates as well as a schematic of an integrated wall-flow 2-way device.[12] The technology consists of coating an NH_3−SCR catalyst within the porous wall of a wall-flow filter (i.e., DPF),
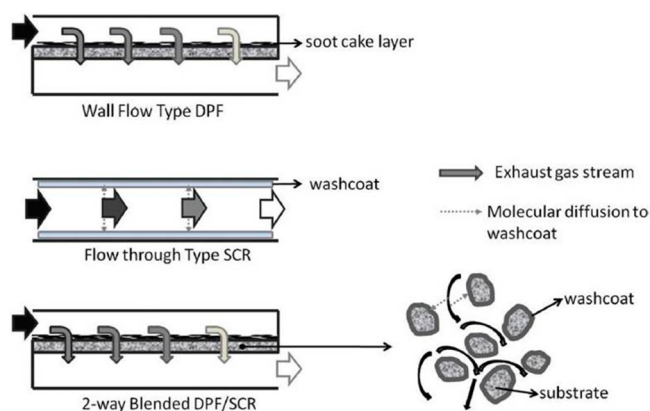

Figure 1. Schematic comparison of a wall-flow DPF, flow-through SCR and integrated wall-flow 2-way DPF/SCR. Reproduced with permission from ref 12. Copyright 2011 Sage Publications.

An integrated SCR/DPF device combines soot filtration with an SCR catalyst, achieving simultaneous soot filtration and NO_x reduction within a single device.[13]

For current conventional aftertreatment strategies, placement of SCR and DPF functionalities in the exhaust stream is governed by SCR activity and soot management. Regeneration of filtered soot requires both heat and oxidant; major oxidants in a diesel application are O_2 and NO_2, with NO_2 oxidizing soot at lower temperature. When exhaust gas temperatures do not reach levels necessary for soot oxidation, active regeneration is necessary; this is facilitated by the injection of fuel in front of the DOC and subsequent oxidation, heating the downstream DPF via the ensuing exotherm to temperature necessary for soot oxidation. For HDD applications, exhaust temperature is comparatively high, and oxidation of soot with NO_2 (i.e., passive soot oxidation) is highly desirable. This motivates placement of the SCR catalyst downstream of the DPF to avoid depletion of NO_2 prior to the DPF; however, SCR catalyst performance is adversely affected by the upstream DPF because of thermal inertia; Koltsakis and co-workers predicted as high as 10% reduced NO_x reduction efficiency due to an upstream DPF.[14] For LDD applications, in which the exhaust gas temperature is comparatively low, resulting in low passive soot oxidation potential, the SCR catalyst is placed upstream of the DPF for improved activity and cold-start performance.

Assuming adequate soot filtration efficiency is achievable with the wall-flow substrate employed, the requirements for successful SCR/DPF integration are maximizing NO_x reduction efficiency while demonstrating acceptable pressure drop across the filter. Maximizing NO_x reduction efficiency will be a function of total SCR catalyst washcoat volume and optimum catalyst coating techniques for maximum SCR efficiency. Demonstrating acceptable pressure drop across the filter will be a function of filter characteristics (e.g., total porosity), catalyst washcoat, and soot management. Thus, high porosity filter materials development is a technology facilitator for integrated SCR/DPF systems for integration of larger catalyst volumes while retaining an acceptable pressure drop.[15−18] The washcoating technique is also a technology facilitator for similar reasons and, thus, is an area of continued optimization by catalyst manufacturers.

This leaves soot management as an area of focus for successful deployment of integrated SCR/DPF technology. For LDD applications that must rely predominantly on active soot

regeneration, soot loading and time between regenerations must be closely controlled because of the exotherm from soot burn and possible adverse effects on SCR catalyst deactivation. In contrast, for HDD applications that rely on significant passive soot oxidation, SCR/DPF integration is more complex. Passive soot oxidation is NO_2-dependent, and current emission control strategies employ oxidation functionality in DPFs for enhanced NO_2 production and improved passive soot oxidation capacity. Integrated SCR/DPF systems will likely not possess oxidation functionality because of its adverse impact on reductant usage (i.e., NH_3 oxidation). In addition, SCR processes will consume NO_2 competitively with soot oxidation. Thus, the incorporation of SCR/DPF for HDD applications must be developed and controlled in such a manner that optimizes NO_2 availability for passive soot oxidation to avoid (or minimize) the fuel penalty associated with the increased active regeneration frequency.

The motivation for integration of DPF and SCR functionalities into a single device resides in both performance and reduced aftertreatment volume. The integration of DPF and SCR functionalities into a single device has the potential to reduce the number of aftertreatment "bricks" required, leading to reduced volume, reduced cost, and increased simplicity. With regard to performance, development of advanced wall-flow DPF substrates have allowed engine manufacturers to focus their attention on reducing the amount of NO_x emitted from tailpipes. Integration of DPF and SCR provides a pathway for NO_x emission reduction with greater flexibility. By integrating SCR and DPF functionalities, the adverse effect of upstream DPF thermal inertia on SCR catalyst performance can be largely mitigated, thereby improving the cold-start NO_x performance.[14] Increased SCR catalyst washcoat volumes are feasible through different configurations, which would provide increased NO_x conversion capability that could facilitate fuel savings by allowing engines to be calibrated to higher engine-out NO_x levels; however, there is uncertainty surrounding the effect of SCR catalyst on passive soot oxidation in integrated SCR/DPF solutions. The adverse impact of upstream SCR on DPF passive soot oxidation is well documented; the extent to which this can be minimized is a topic of significant current interest which this paper discusses.

A number of early works have been performed to evaluate the feasibility of simultaneous NO_x and PM aftertreatment with integrated SCR/DPF systems.[19−23] These early works successfully demonstrated the feasibility of combined NO_x and PM reduction and identified key barriers to its effective deployment. Some of these key technology hurdles include

- Increased back pressure resulting from high SCR washcoat volumes
- Necessity of improved washcoating technology for maximizing SCR catalyst volume and gas contact
- Soot oxidation challenges

Washcoat optimization is a topic to which many SCR/DPF investigators have alluded;[24] however, little has been mentioned of the details of the SCR catalyst integration within the wall-flow filter. This work attempts to provide insight into the impact of SCR catalyst location on soot-loading characteristics of the filter. The intent is to improve the level of understanding for maximizing SCR catalyst washcoat volumes while minimizing the back pressure that results from soot collection.

The impact of soot on SCR reactor performance has been reported by many with conflicting results in some cases. This work will report on the impact of soot on NO_x conversion performance to develop a more fundamental understanding of the nature of the interaction. In addition, as previously mentioned, the adverse effect of SCR reaction on passive soot oxidation (with NO_2) is a recognized barrier to successful SCR/DPF integration for HDD. However, detailed understanding of the nature of this interaction and the primary reaction mechanisms that govern system performance are lacking. This work will report on the impact of SCR reaction on passive soot oxidation in an attempt to develop a more fundamental understanding of the primary reactive drivers that govern soot oxidation feasibility in the integrated system.

2. EXPERIMENTAL METHODS

High porosity cordierite wall-flow filters (i.e., DPFs; 25 mm o.d. × 40 mm long) were coated with SCR catalyst for the purpose of this investigation. The SCR catalyst employed was a Cu-chabazite catalyst loaded by an unnamed industrial partner to varying catalyst density. Upon receipt, samples were degreened under lean conditions at 10% O_2, 5% H_2O, balance N_2.

Soot loading and reactive interrogation of the SCR/DPF samples occurred separately in iterative fashion. The samples were loaded with soot employing the exhaust from a 2003 VW Jetta TDI following a DOC placed upstream of the filters. During soot loading, the exhaust was continually measured with a smoke meter to determine post-DOC particulate density. A slip stream of the exhaust was pulled through the SCR/DPF sample with the use of a vacuum pump, with the slip stream subsequently conditioned and measured for flow rate. Flow rate was electronically controlled to target 55 000 GHSV during loading; measured particulate density and flow rate were combined to target a desired total soot loading, typically 4 g/L. Pressure drop was measured at ports just upstream and downstream of the SCR/DPF sample holding apparatus. Subsequent to soot loading, the samples were removed and weighed at elevated temperature for redundant measurement of soot mass uptake. Estimated soot loading from smoke meter and mass measurements were in good agreement with typically less than 10% variance.

Reactive measurements were performed on a dedicated test bench designed to accommodate the soot-loaded filter and housing. The simulated exhaust composition, consisting of 9% O_2, 9% CO_2, 8% H_2O, 500−1500 ppm of NO_x (NO and NO_2), and balance N_2, was blended together to simulate post-DOC heavy-duty lean burn diesel exhaust. For measurements including SCR reaction, NH_3 was included at 500−1500 ppm.

Dry gases were supplied by electronically controlled MKS mass flow controllers. Air, N_2, and CO_2 were combined as dry gases and preheated to the desired temperature with an electronically controlled tube furnace. Simultaneous to preheating, the feed stream was humidified to the desired level with an electronically controlled water vaporizer employing a N_2 sweep flow. Following preheating, NO, NO_2, and NH_3 were supplied separately as dry gas feeds to the exhaust flow in desired quantities. The full simulated exhaust was then fed to the reactor assembly consisting of a quartz tube containing the SCR/DPF sample held in place by high temperature alumina paper. An electronically controlled tube furnace controlled the reactor temperature to the desired level. Temperature measurements were made employing Watlow type K thermocouples placed just upstream and downstream of the DPF/SCR sample. Pressure drop measurement was made via ports located just upstream and downstream of the sample holder. Gas analysis

was accomplished with a Nicolet 6700 FTIR employing a metal gas cell heated to 190 °C. Sample gas transfer lines to the FTIR were heated to 200 °C to avoid NH_4NO_3 formation. FTIR gas analysis was conducted at 100 Torr through the use of a vacuum pump located downstream from the FTIR gas cell and an electronically controlled MKS pressure controller. Redundant NO_x measurement was made via a heated California Analytics chemiluminescent NO_x meter. Control of the entire assembly, including mass flow controllers, all heated zones, and data acquisition, was facilitated by a custom designed Visual Basic control system.

Bench-scale reactive measurements were performed in pseudosteady state fashion by employing slow thermal ramping, ~2 °C/min, from ~200 to 550 °C. Reactive measurements included SCR performance for NO_x conversion efficiency and temperature-programmed oxidation (TPO) for soot oxidation characterization.

Scanning electron microscopy (SEM) was performed with a JEOL 5900 scanning electron microscope. The sample was cut perpendicular to the cordierite channels with a wet diamond wafer saw, potted in LR white resin, and spun at 2000 rpm for 10 min in a standard centrifuge and subsequently evacuated to remove gases from the filter and resin. Following polymerization and hardening of the resin, the sample was cross-sectioned perpendicular to the channel axis, ground and polished with a series of diamond grits (25, 9, 3, and 1 μm), and final polished with 0.05 μm γ-alumina.

Pore characteristics (porosity, pore size distribution) were measured via Hg intrusion employing a Micromeritics AutoPore IV 9500 series mercury porosimeter.

3. RESULTS AND DISCUSSION

SEM imaging and porosity characteristics were compared with soot collection characteristics in an attempt to determine the combined effect of catalyst loading and location on filtration behavior. Figure 2 shows the SEM imaging of the uncoated DPF substrate (A) and SCR/DPF samples coated with ~60 (B), ~90 (C), and ~150 g/L (D) SCR catalyst loading density. In each of the subfigures, using A as a reference, black is background or void space, (off-)white is cordierite substrate, and gray is SCR catalyst. Since channels are plugged at
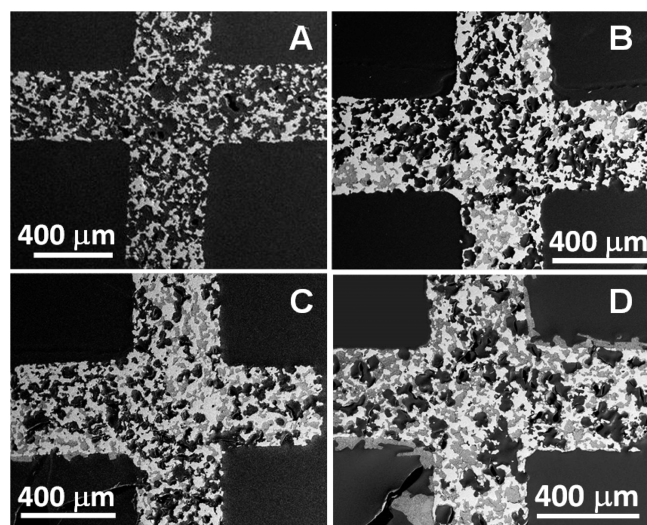

Figure 2. SEM imaging of clean DPF (A) and SCR/DPF samples coated with 60 (B), 90 (C), and 150 (D) g/L SCR catalyst loading.

alternating ends on the filter, channels in opposing corners of each subfigure in Figure 2 are common to a respective end of the filter; direction of exhaust flow through the samples is reversed by simply rotating the filter axially with respect to flow.

With the SCR/DPF sample loaded to 60 g/L catalyst density (B), SCR catalyst is deposited solely within the filter wall microstructure and not on the channel walls. Catalyst is located heavily to one side of the filter wall and penetrating, on average, 40−60% across the width of the wall. In the SCR/DPF sample loaded to 90 g/L catalyst density (C), SCR catalyst is similarly deposited predominantly within the filter microstructure and also heavily to one side of the filter wall. Periodic occurrences of the catalyst penetrating the full width of the porous filter wall are evident, and with little or no catalyst present on either set of the channel walls. Comparatively in the SCR/DPF sample loaded to 150 g/L catalyst density (D), there are notable differences to the previous two samples. First, catalyst of varying amounts is present on one set of channel walls, outside of the wall microstructure. And second, the SCR catalyst consistently penetrates across the full width of the filter wall; thus, catalyst is in intimate contact with both filter walls. It is worth noting that the SEM imaging was conducted at four locations within the coated samples, with the images shown above selected randomly from the samples analyzed. The results discussed in the text with regard to catalyst location were consistently reflected in all samples analyzed.

Figure 3 shows the corresponding results of Hg porosimetry analysis of the DPF and SCR/DPF samples, presented as
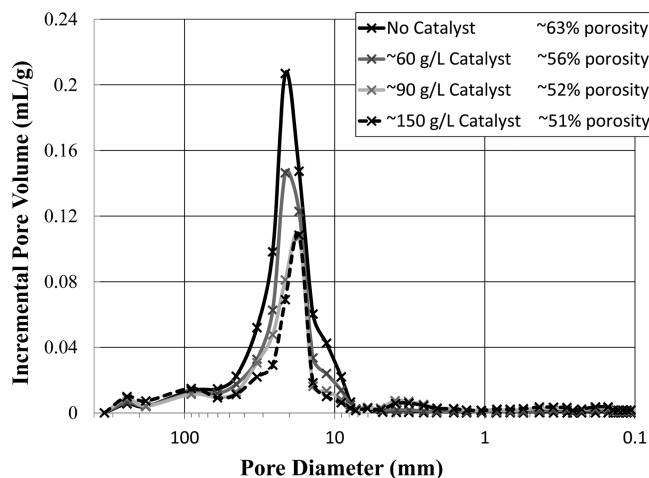

Figure 3. Hg porosimetry analysis of DPF (no catalyst) and SCR/DPF samples loaded to 60, 90, and 150 g/L SCR catalyst.

incremental pore volume (mL/g) versus pore diameter (mm). The uncoated DPF measured 63% porosity. The addition of 60 g/L of SCR catalyst decreased the total porosity to 56%, with similar peak pore diameters for both the DPF and 60 g/L SCR/DPF sample. This, coupled with SEM imaging, provides good indication that the 60 g/L of SCR catalyst was depositing solely within the filter wall microstructure. Similar peak pore diameters support the observation that a large portion of the porous filter wall remained largely void of catalyst.

With an additional 30 g/L catalyst (i.e., 90 g/L total SCR catalyst), the total porosity decreased to 52% with a slight shift in the peak pore diameter to a smaller maximum. The shift in the peak pore diameter suggests that the less porous filter wall is void of catalyst versus the 60 g/L sample. These results
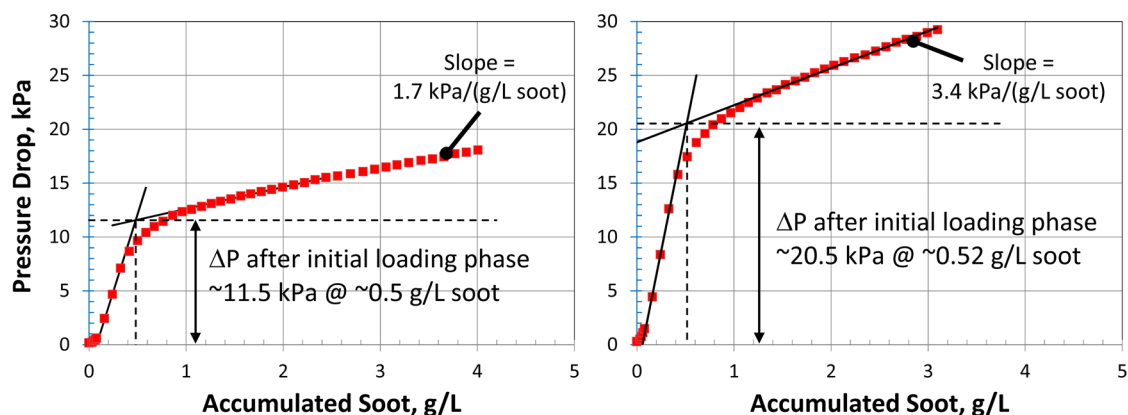
Figure 4. Soot-loading characteristics of 90 (left) and 150 g/L (right) SCR/DPF samples configured such that catalyst was present predominantly on the upstream portion of the filter microstructure and on the inlet channel wall (for the 150 g/L sample).
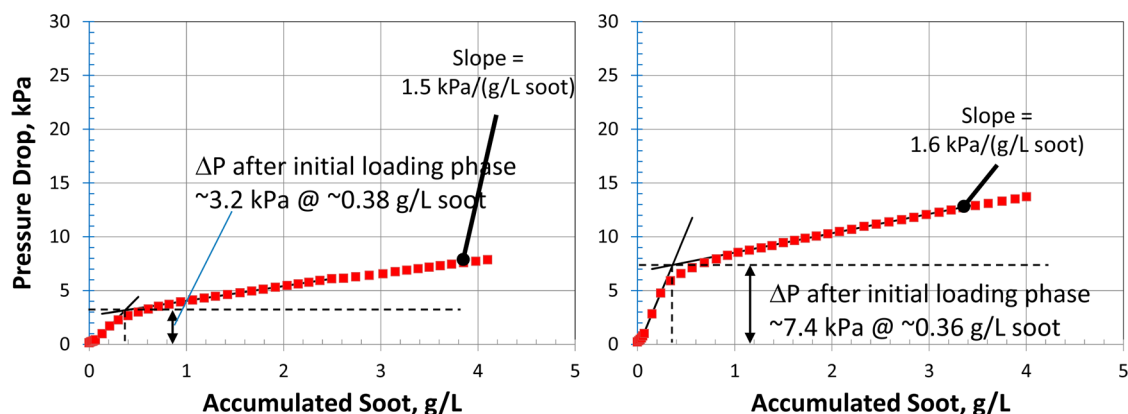

Figure 5. Soot-loading characteristics of 90 g/L (left) and 150 g/L (right) SCR/DPF samples configured such that catalyst was present predominantly on the downstream portion of the filter microstructure and on the outlet channel wall (150 g/L sample).

coupled with SEM indicate that 90 g/L of SCR catalyst continued to deposit predominantly within the filter wall microstructure.

With an additional 60 g/L catalyst (i.e., 150 g/L total SCR catalyst), the total porosity did not decrease significantly but, rather, remained fairly constant at 51%, with no change in the peak pore diameter (versus 90 g/L). The SCR catalyst filled a small number of very large pores >20 mm, leading to a slight asymmetric narrowing of the pore distribution. One difference from the previous two samples is that a significant amount of catalyst did not penetrate the wall microstructure but, rather, remained coated on the filter channel.

In summary, total porosity and peak pore characteristics, coupled with SEM imaging, support the notion that up to 90 g/L SCR catalyst was loaded into the filter wall microstructure, penetrating the width of the porous wall to varying degrees but loaded predominantly heavy to one side of the filter wall. Comparatively, the results suggest that >90 g/L SCR catalyst was largely deposited as a coating on the filter channel, with the catalyst deposited typically across the full width of the porous filter wall.

3.1. Soot-Loading Behavior. The 90 and 150 g/L SCR/DPF samples were loaded with soot to determine the effect of catalyst location on the soot loading characteristics of the samples. Figures 4 and 5 show on-engine backpressure (i.e., pressure drop) resulting from the 90 g/L sample (left) and the 150 g/L sample (right) as they are loaded with soot. In Figure 4, samples are configured with the catalyst on the upstream

portion of the filter (in close proximity to the collected soot). Figure 5 shows analogous backpressure measurements with the samples configured in the opposite direction (i.e., catalyst on the downstream portion of the filter). The data presented here were very reproducible, with subsequent pressure drop (versus collected soot) traces in close agreement.

The pressure drop of the clean SCR/DPF samples was comparatively low: 0.35 and 0.44 kPa for 90 and 150 g/L SCR catalyst loading, respectively, under the conditions tested. Thus, collected soot affected filter permeability much more significantly than the catalyst washcoat; however, both the catalyst concentration and location impacted the magnitude of pressure drop resulting from the collection of soot. The most prominent effect occurred during depth filtration of soot; this is not surprising, given the knowledge that the catalyst is largely located within the wall microstructure. With the catalyst on the upstream portion of the filter (Figure 4), the system demonstrated 11.5 and 20.5 kPa back pressure for the 90 and 150 g/L SCR catalyst densities, respectively, under the conditions tested when it transitioned from depth to cake filtration mode. Comparatively, with the catalyst on the downstream portion of the filter (Figure 5), the system demonstrated 3.2 and 7.4 kPa back pressure for the 90 and 150 g/L SCR catalyst densities, respectively, under the conditions tested when it transitioned from depth to cake filtration mode.

The pressure drop during depth filtration is affected by the amount of SCR catalyst located on the upstream portion of the filter wall microstructure. The magnitude of pressure drop does

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