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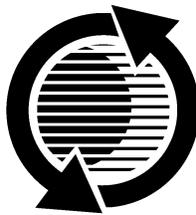Oak Ridge National Lab.

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ABSTRACT

There is a need for an efficient, durable technology to reduce NOx emissions from oxidative exhaust streams such as those produced by compression-ignition, direct-injection (CIDI) diesel or lean-burn gasoline engines. A partnership formed between the DOE Office of Advanced Automotive Technology, Pacific Northwest National Laboratory, Oak Ridge National Laboratory and the USCAR Low Emission Technologies Research and Development Partnership is evaluating the effectiveness of a non-thermal plasma in conjunction with catalytic materials to mediate NOx and particulate emissions from diesel fueled light duty (CIDI) engines. Preliminary studies showed that plasma-catalyst systems could reduce up to 70% of NOx emissions at an equivalent cost of 3.5% of the input fuel in simulated diesel exhaust. These studies also showed that the type and concentration of hydrocarbon play a key role in both the plasma gas phase chemistry and the catalyst surface chemistry. More recently, plasma/catalyst systems have been evaluated for NOx reduction and particulate removal on a CIDI engine. Performance results for select plasma-catalyst systems for both simulated and actual CIDI exhaust will be presented. The effect of NOx and hydrocarbon concentration on plasma-catalyst performance will also be shown.

INTRODUCTION

There is a large PNGV effort to develop the "next generation" of vehicles that can achieve 80 mpg fuel economy while simultaneously meeting emission standards for NO_x, CO and hydrocarbons. New hybrid CIDI (compression ignition direct injection) diesel fueled engines can meet the fuel economy requirements, however, NO_x emission standards can not be simultaneously met with currently available technology. Current after-treatment devices can not reduce NO_x sufficiently in lean burn (net oxidative) exhaust typical of diesel exhaust (1). Variations of combustion conditions can lower NO_x emissions, however, particulate emissions increase and there can also be a concomitant fuel efficiency loss. A viable "lean burn" NO_x after-treatment technology would enable the use of CIDI and lean burn gasoline engines, resulting in significant increases in combustion efficiency of fossil fuels in both the transportation sector and the utility sector, which combined are responsible for 66% of CO_2 emissions in the United States.
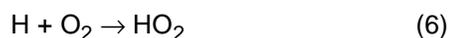
The central scientific challenge to meeting NO_x emission standards in diesel or lean burn gasoline exhaust streams is to promote the reduction reaction of NO_x to N_2 and O_2 in an oxidizing atmosphere. There has been considerable activity in the last 10 years concentrated on developing new materials for lean NO_x reduction. While Cu-ZSM-5 and other zeolites have shown promise for NO_x reduction in simulated vehicle exhaust, practical application of these materials is precluded due their narrow temperature window for NOx conversion, poor hydrothermal stability, and poisoning from sulfur in the fuel. (2). In addition, passenger cars operated on low-speed driving cycles do not generate high enough exhaust temperatures to obtain high NOx conversion efficiencies. These catalysts utilize unburned hydrocarbons present in the exhaust to reduce NO_x by

selective catalytic reduction (SCR). Likewise, SCR catalysts have been developed that can reduce NOx if ammonia (as urea) is added to the exhaust stream as a reductant. This technology would require new infrastructure to supply the urea and assumes that the consumer would reliably refill the urea tank. NOx storage catalysts have also shown high reduction activities, however, these materials become poisoned by sulfur in the exhaust after relatively short periods of time (3). Recent work on non-thermal plasma/catalyst systems has shown significant NO_x reduction activity for simulated lean burn and diesel engine exhaust (4,5). In these systems, the plasma creates energetic electrons and free radicals which interact with gas phase molecules to promote NO_x reduction over a catalyst placed in or downstream from the plasma.

In order to design new plasma catalyst systems with improved NOx reduction efficiency, it is important to understand the chemistry that occurs in both the gas phase of the plasma and over the catalyst surfaces. The current understanding of the plasma chemistry can be found in the literature (4-7). In summary, the NO_x chemistry in the plasma is dominated by the hydrocarbon, water, and oxygen components. As shown by Penetrante, NO is oxidized to NO_2 primarily by:

$$NO + HO_2 \rightarrow NO_2 + OH \qquad (1)$$

$$NO + RO_2 \rightarrow NO_2 + RO \qquad (2)$$
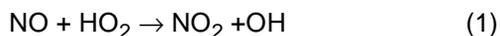
where R is a hydrocarbon radical (7). Electron-impact dissociation of water produces additional OH radicals. HO_2 is produced from reaction of O_2 with hydrocarbon intermediates (7). For example:

$$CH_2OH + O_2 \rightarrow CH_2O + HO_2 \qquad (3)$$

$$CH_3O + O_2 \rightarrow CH_2O + HO_2 \qquad (4)$$

$$HCO + O_2 \rightarrow CO + HO_2 \qquad (5)$$

$$H + O_2 \rightarrow HO_2 \qquad (6)$$
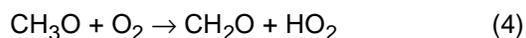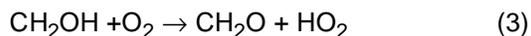
Oxygen and hydroxyl radicals produced by electron-impact dissociation and reaction of NO with HO_2 are consumed primarily by reactions with the hydrocarbons (7). Exhaust components such as Ar, CO_2, CO and H_2 do not significantly influence the gas phase chemistry (8). Several important conclusions can be drawn from the information on the lean mix gas phase chemistry. First, the dominant NO_x chemistry is oxidation of NO to NO_2. Second, gas phase chemistry alone cannot result in significant reduction of NO to N_2; this can only be accomplished with a heterogeneous catalyst. Finally, activity testing of plasma/catalyst systems in the absence of either hydrocarbon or water is not meaningful.

Many catalysts that show activity for lean NOx reduction exhibit diminished or zero activity in combination with a plasma. Therefore, there is a great need to develop new catalysts that are active when combined with a plasma device. A previous study by Balmer et al (9) measured the activity of a number of oxides when combined either in or downstream from a non-thermal plasma. In summary, oxides such as borosilicate glass, zirconia, and barium titanate do not significantly change the gas phase NO_x chemistry when combined with a plasma. Gas phase reactions dominate, resulting in the oxidation of NO to NO_2. However, heterogeneous reactions on Cu-ZSM-5, γ-Al_2O_3, and Na-ZSM-5 and TiO_2 do influence the NO_x chemistry. Cu-ZSM-5 primarily acts to convert NO_2 back to NO at 180°C. Na-ZSM-5 also converts a portion of NO_2 back to NO, but, in addition, exhibits 35-40% apparent NO_x reduction at 180°C. Activated γ-Al_2O_3 with a surface area of 75 m^2/g reduces 40% NO_x at 180°C. Activated gamma alumina from a different vendor with a higher surface area (200 m^2/g) did not show any appreciable plasma assisted NOx conversion. The anatase form of titania reduces up to 35% of NOx at 150J/L when the catalyst is contained in the plasma region, but does not show any appreciable conversion when placed downstream from the reactor. This phenomenon was attributed to photoactivation of anatase in the plasma (9).

Vogtlin et al. (10) showed that a wire-in-tube corona device followed by gamma alumina reduces up to 80% of NOx at 400°C in a simulated lean exhaust containing 500 ppm NO, 1000 ppm C_3H_6, 10% O_2, 10% CO_2, 5% H_2O, and balance N_2 at an input power of 5 J/L. Shimizu et al. (11) reported that a "bare" coaxial silica tube barrier-type discharge reduced from 10-20% of NO_x over a temperature range from 100-500°C in a gas mix of 400 ppm NO, 2.5% CO_2, 2.5% O_2 and N_2. When a Pd/Rh three-way catalyst, Cu-ZSM-5, and Na-ZSM were introduced into the plasma, 50-60% NO_x reduction was reported at input energies of 100J/L (11).

EXPERIMENT

Bench scale studies were performed using a dielectric barrier packed bed reactor described in Tonkyn et al. (12) and Balmer et al. (9) as well as a proprietary reactor design. Unless otherwise noted, the feed gas contained the gas mixture and concentrations listed in Table 1. The composition in Table 1 simulates diesel exhaust with a small amount of hydrocarbon injection. The product gases were analyzed with a chemiluminescent NO_x analyzer (CLA), a mass spectrometer (MS), and a Fourier Transform Infrared Spectrometer (FTIR). Catalysts were tested in the discharge region in a dielectric barrier packed bed reactor (single-stage) or downstream from the region in which the discharge occurs (two-stage) (9).

The power deposited into the reactor was measured with a capacitive circuit and a high voltage probe for bench tests (12). The beta (β) parameter, which is used to characterize the energy efficiency of the discharge treatment, is represented as follows:

$$[NO_x] = [NO_x]_f + ([NO_x]_o - [NO_x]_f) * e^{-E/\beta} \qquad (7)$$

Where β is the first order decay parameter in Joules/ standard liter, $[NO_x]_o$ is the initial NO_x concentration and $[NO_x]_f$ is the final limiting NO_x concentration.

Table 1. Concentrations of components in simulated exhaust gas mixture.

Component	Concentration
NO	200 ppm
C_3H_6	525 ppm
C_3H_8	175 ppm
H_2O	7%
O_2	8%
CO_2	7%
Ar	9000 ppm
CO	400 ppm
H_2	130 ppm
N_2	Balance

Engine tests were performed at Oak Ridge National Laboratory using a 1996 1.9L Volkswagen TDI diesel fueled engine. Certification Diesel fuel with a sulfur level of 350 ppm was used for engine testing. The plasma device was a double dielectric barrier design with alumina barrier and copper electrodes. The catalyst was placed downstream from the region in which the plasma was generated, similar to the 2-stage design presented in Balmer et al. (9) Catalyst B was coated onto 300 cell/inch cordierite monoliths provided by DaimlerChrysler Corporation.

RESULTS AND DISCUSSION

BENCH TESTS WITH ZEOLITIC CATALYST B – Focused efforts by PNNL and the Low Emissions Technologies Research and Development Partnership have lead to the discovery of new catalyst formulations that reduce NOx when placed in or downstream from a non-thermal plasma. Figure 1 shows the NOx conversion as a function of energy density for a new catalyst, designated catalyst B. It can be seen that a "green" (fresh) catalyst converts 85% NOx and that after aging in simulated vehicle exhaust for 120 hours (two-stage) it converts 75% NOx. This experiment was run at 180°C ,a space velocity of 12,000 hr^{-1} and gas composition: 150 ppm NO, 450 ppm C_3H_6, 396 ppm CO, 6% O_2, 2% H_2O and 11% CO_2.

The exact composition of this catalyst is currently proprietary information. The conversion of NO to NO_2 (not shown) is close to 100% prior to the catalyst. The portion of NO_2 that is not reduced is converted back to NO over the catalyst surface.

It has been shown that the dominant NOx chemistry in the plasma, which is conversion of NO to NO_2, is dominated by the hydrocarbon, water, and oxygen components (7,9). Exhaust components such as Ar, CO_2, CO and H_2 do not significantly influence the gas phase chemistry (8). In addition, previous work has shown that some oxygen and propylene are required for complete oxidation of NO to NO_2 in the plasma (without catalyst or with inert material) (7,9). Maximum NO to NO_2 conversion occurred over inert borosilicate glass beads when gas mixtures contained at least 2% oxygen or at least 1000 ppm propylene (at 4:1 propylene:NO) at 100-300°C. Gas phase chemistry alone cannot result in significant reduction of NO to N_2; this can only be accomplished with a heterogeneous catalyst (7,9).
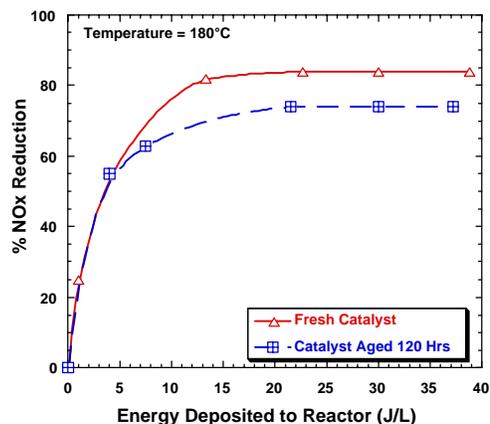

Figure 1. NOx conversion as a function of energy density (two-stage) for a proprietary catalyst that is fresh and aged 120 hours in simulated exhaust.

The effect of propylene concentration on NOx reduction in the presence of catalyst B was measured at 215°C at a constant power of 20 J/L in a 2-stage configuration (Figure 2). The gas mixture contained 178 ppm NO, 381 ppm CO, 1.8% H2O, 8.3% CO_2, 5.7% O_2, with a nitrogen balance. The plasma temperature was 131°C and the catalyst temperature was 215°C. NO is almost completely converted to NO_2 in the plasma. There is a steep increase in NO_x reduction activity up to 350 ppm of propylene or an equivalent of 1050 ppm C_1 ($\approx$6:1 $C_1:NO_x$). Additional propylene does not enhance NOx reduction activity. The NO conversion curve shows that there is a portion of NO_2 that is converted back to NO over the catalyst. The concentration of propylene required to reach maximum NOx reduction over catalyst B correlates well with the amount of propylene required to completely convert NO to NO_2 in the plasma. Indeed, studies published elsewhere on catalyst B show that it is more active for NO_2 reduction than for NO reduction (13). When considering the ratio of propylene to NOx, the ratio required to reach the maximum reduction over the catalyst is slightly higher than for complete conversion of NO to NO_2 in the plasma.
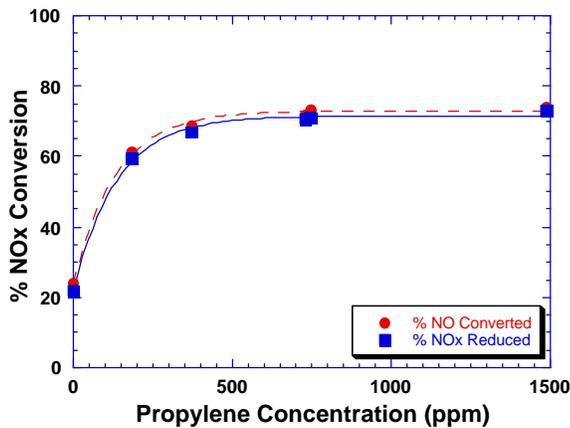
Figure 2. Percent NOx conversion over catalyst B as a function of propylene concentration at 215 °C.

The effect of increasing space velocity on this catalyst was examined at a constant power under the same reaction conditions. As shown in Figure 3, there is no change in activity when the space velocity is increased from 10,000 hr^{-1} to 18,000 hr^{-1}. There is less than a 7% decrease in activity when the space velocity is increased from 18,000 hr^{-1} to 27,000 hr^{-1}.
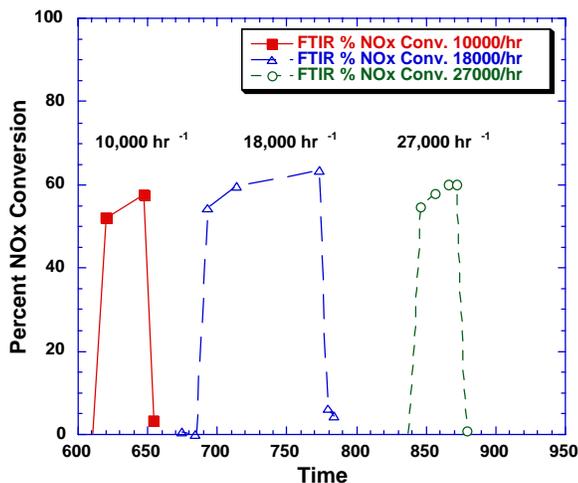

Figure 3. Percent NOx conversion over catalyst B at a constant power (27 J/L) for three different space velocities; 10,000 hr^{-1}, 18,000 hr^{-1}, and 27,000 hr^{-1}.

The NOx reduction activity as a function of temperature was measured over the range of 150-370°C. As shown in Figure 4, the NOx reduction activity of catalyst B ranges from 67-75% between 150-300°C then decreases to 55% at 370°C. The operational temperature range of this catalyst coincides well with the expected exhaust temperatures from light-duty compression ignition direct injection engine exhaust.
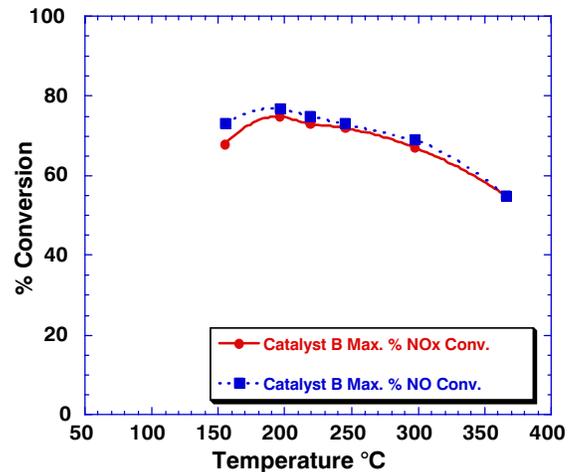

Figure 4. Percent NO and NOx conversion as a function of temperature for catalyst B.

In an attempt to account for any nitrogen-containing by-products that could go undetected by the CLA and to obtain a complete nitrogen balance, activity tests were performed where N_2 in the simulated exhaust was replaced with He. In addition, CO and CO_2 were removed from the mix. Previous work has shown (8), and experiments confirmed that there is not a significant change in the product chemistry when He replaces N_2 and when CO and CO_2 are removed. Approximately 80% of the total nitrogen could be accounted for downstream of catalyst B using FTIR and Gas Chromatograph analysis. However, if an oxidation catalyst (0.5% Pt on Al_2O_3 from Aldrich) was placed downstream from catalyst B, 100% of the nitrogen could be accounted for in the form of N_2 and N_2O. Therefore, some intermediate product which is formed after catalyst B is converted to N_2 or N_2O by a Pt catalyst. The ratio of N_2O to total nitrogen species formed as a function of temperature is shown in Figure 5 for catalyst B followed by the Pt on Al_2O_3 catalyst. Nitrogen is the favored product over the range of temperatures tested, however, some N_2O is also formed at all temperatures. Nitrous oxide production accounts for as much as 40% of the total nitrogen at 140°C, then decreases with increasing temperature. At 200°C and 280°C respectively nitrous oxide accounts for only 13% and 8% of the total nitrogen.

Catalyst B was analyzed for surface adsorbed nitrogen-containing species by temperature programmed desorption with mass spectrometer analysis and by wet chemical techniques. Both of these techniques showed that a sample of catalyst B used for 58 hours contained 0.9 $cc_{(Nox)}/g_{(solid)}$. During this period the catalyst reduced a total of 88 $cc_{(Nox)}/g_{(solid)}$. Therefore, only 1% of the total NOx removed is adsorbed to the surface. It has not been determined whether this adsorbed NOx is an active intermediate in the NOx reduction reaction or whether it is adsorbed to a site that does not participate in NOx reduction.
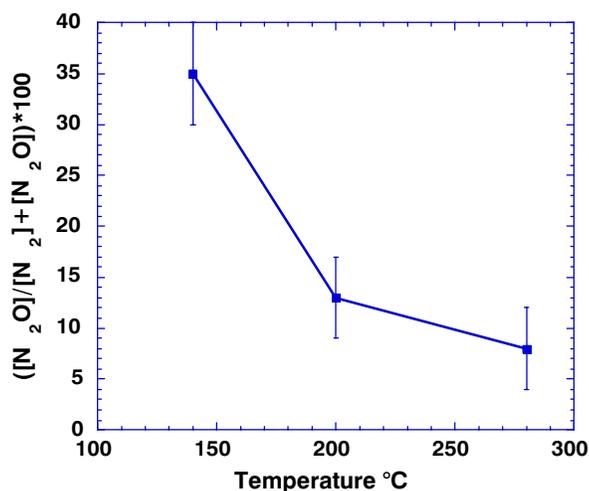
Figure 5. The ratio of nitrogen to nitrous oxide formed downstream from catalyst B and a 0.5% Pt on Al_2O_3 catalyst.

Sulfur dioxide present in the exhaust gas from diesel-fueled engines has been shown to rapidly poison NOx storage catalysts, and can likewise have a deleterious effect on many lean-NOx catalysts. In order to determine if sulfur dioxide would degrade the performance of catalyst B, 50 ppm of sulfur dioxide was added to the simulated exhaust gas mix. This concentration, which is higher than sulfur dioxide levels expected for diesel exhaust, was chosen to accelerate any poisoning effects. During a 30-hour test in a single stage configuration, no decrease in NOx reduction activity was observed. The NOx reduction activity as a function of energy density before and after exposure to 50 ppm sulfur for 30 hours is shown in Figure 6. Note that this plasma/catalyst system will need an oxidation catalyst downstream in order to remove remaining HC and aldehydes; this downstream catalyst will have some sulfur sensitivity, so the result presented here should not be taken as evidence of high sulfur tolerance of a future vehicle system.

ENGINE TESTS WITH CATALYST B – Engine tests were performed at Oak Ridge National Laboratory in Oak Ridge, TN at the Advanced Transportation Technology Center. PNNL bench-scale devices were scaled-up to treat the full exhaust stream from a 1996 Volkswagon 1.9 liter TDI diesel-fueled engine. The engine conditions used to collect NOx reduction data for the device are described in Table 2. The concentrations of oxygen, CO_2, CO, hydrocarbon, and NOx in the exhaust were measured using a combination of analytical devices including a Fourier transform infrared spectrometer, a chemiluminescent NOx analyzer (CLA), and a flame ionization detector. The engine-out concentrations of these components are shown in Table 3. Because the plasma volume was much smaller than the volume of the catalyst bricks, the space velocity through the catalysts was 12,500 hr^{-1} while the space velocity through the plasma was 275,000 hr^{-1}.
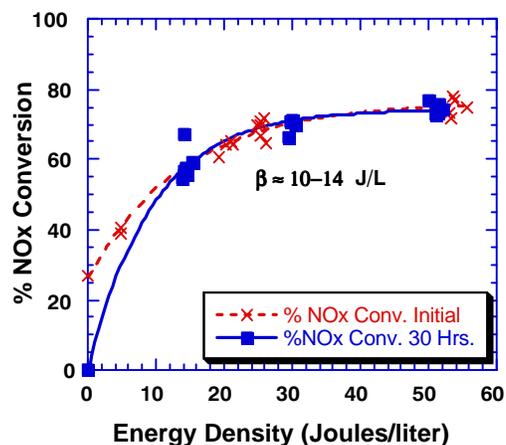

Figure 6. NOx conversion as a function of energy density for catalyst B before exposure to SO_2 and after exposure to SO_2 for 30 hours.

Table 2. Engine Conditions

Revolutions per Minute (rpm)	1900
Torque (ft•lb)	61
Horse Power (HP)	22
Air/Fuel Ratio	28
Air Flow (scfm)	59-63
Fuel Flow (cc/sec)	1.3
Temperature into Device (°C)	276-279
Temperature out of Device (°C)	171-215

Table 3. Engine Out Exhaust Gas Concentrations

Oxygen	11%
Carbon Dioxide	7%
Carbon Monoxide	150 ppm
Hydrocarbon as C1	140 ppm
NOx	320 ppm

Figure 7 shows the NOx conversion as measured by: 1.) the chemiluminescent NOx analyzer (NOx that does not show up as NO or NO_2 is assumed to be reduced), and 2.) the FTIR (NOx that is not detected as N_2O, NO_2, NO or other nitrogen-containing species is assumed reduced to N_2). The background value is the amount of NOx that was "removed between the inlet and outlet of the device. The background may arise from thermal conversion, adsorption, or calibration differences between the inlet and outlet CLAs. The "corrected" FTIR value takes into account a small amount of drift in engine-out NOx concentration that occurred over the testing period. It can be seen from the Figure that with no added hydrocarbon (engine out C_1:NOx = 0.4:1) there is no significant reduction of NOx. For this data point, there was a large discrepancy between the CLA and the FTIR. The FTIR data is considered more accurate since the NOx concentration as measured by the CLA can be affected by other components in the exhaust gas (14).

When 870 ppm C_3H_6 (2610 ppm C_1) is added to the exhaust, 47-57% NOx reduction is measured. There is better agreement between CLA and FTIR data for the second data point (taken at 2 hours with added hydrocarbon). The CLA failed after 2 hours of testing. All data in Figure 7 points were taken at an apparent power of 3.2 KV*amps. The overall conversions observed in engine testing are considerably lower than those observed in bench tests for the same hydrocarbon concentrations. Some differences may be due to the fact that the bench tests were performed using solid catalyst extrudates rather than coated monoliths. Optimization of processing procedures is expected to increase activity of coated monoliths.
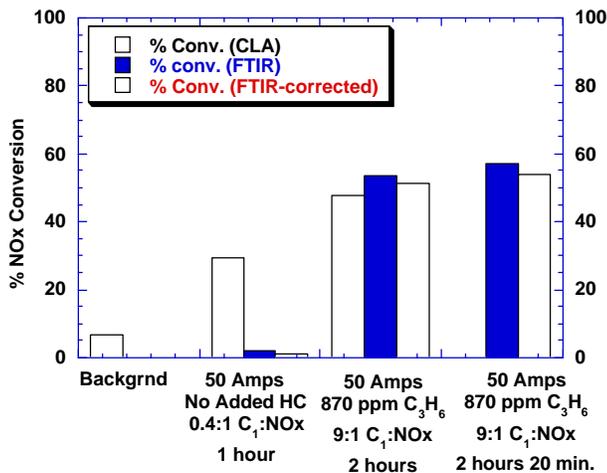

Figure 7. NOx reduction from diesel engine exhaust for a 2-stage plasma catalyst system with and without added propylene.

Apparent power (volts*amps) used by the plasma reactor was measured at the line supply to the high voltage circuit (wall plug) using a standard volt-meter and a clamp-on ammeter. It is important to note that this measurement does not reflect the amount of power delivered to the plasma reactor and that no attempts have been made to minimize losses through the power supply for this first prototype system. While losses through the full-scale system are not known, for bench-scale systems, the true power delivered to the reactor is typically 50% of the wall plug power. Figure 8 shows the NOx conversion from the CLA and FTIR as a function of the volt*amps with added propylene at a ratio of 9:1 C_1:NOx and a space velocity of 12,500 hr^{-1}. The maximum conversion occurs near 50 amps and 64 V which corresponds to 4 horsepower or 18% of the engine-out horsepower. This energy consumption is clearly too high to be practical, however, we expect that with optimization of the power delivery system, the power consumed will be greatly reduced. The form of the conversion vs. power is similar to the lab scale results, suggesting that the delivered power required is of the order of 15 J/L or 0.6 HP (2.7% engine-out).
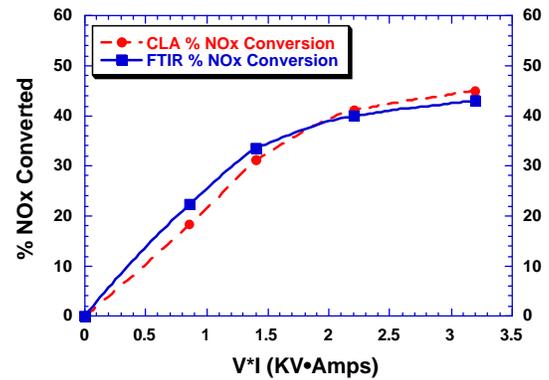

Figure 8. NOx conversion vs. apparent power as measured going into the power supply (wall plug).

The amount of propylene added to the exhaust was varied in order to determine the minimum hydrocarbon needed to reach the maximum NOx reduction. Figure 9 shows the NOx reduction for 140-2880 ppm total C_1 (3:1 to 9:1 C_1:NOx). Total HC into the plasma device consisted of 140 ppm C_1 from engine-out exhaust plus added propylene to achieve a higher carbon to NOx ratio. It can be seen that the maximum NOx conversion is achieved at a C_1:NOx ratio of 6:1 and that no additional benefit is obtained by increasing the ratio to 9:1. This result is in good agreement with bench data on catalyst B which showed that maximum NOx conversion can be obtained at a C_1:NOx ratio of 6.
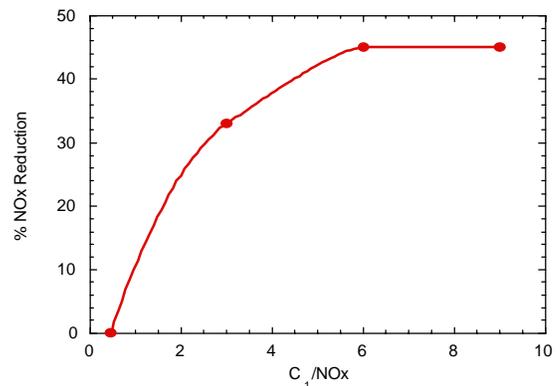

Figure 9. % NOx reduction as a function of the ratio of C_1/NOx in the exhaust. Engine-out C_1 was 140 ppm and additional C_1 was achieved with added propylene.

The hydrocarbon speciation exiting the plasma and catalyst with propylene addition was measured by FTIR. The propylene that is not completely oxidized to CO_2 is primarily converted to carbon monoxide, formaldehyde and acetaldehyde.

After two days of testing, the test point shown in Figure 7 (2 hours of testing at 9:1 C_1:NOx) was duplicated to

determine if the NOx reduction efficiency had degraded with time. The corrected concentration of NOx reduced from the FTIR had decreased from 53% to 48% after this two day period. It is not clear from the initial data if this drop in activity is due to catalyst degradation or if it is within the error limits of the test. Laboratory tests are currently being performed to determine if catalyst deactivation occurs, and if it does, if damage to the catalyst is permanent or reversible (such as surface coking).

CONCLUSION

A two-stage plasma catalyst system developed by PNNL and the LEP was tested in simulated diesel exhaust and in actual diesel exhaust from a VW 1.9 liter, TDI engine. In simulated exhaust the system could achieve 70% NOx reduction for extended periods of time with 50 ppm SO_2 in the gas mixture. Optimum NOx reduction was achieved between the temperatures of 150-300°C, at a C_1 to NOx ratio of 6 and at an input power of 10 J/L. With a platinum catalyst following the plasma reactor, a complete nitrogen balance can be obtained. The nitrogen-containing product distribution is temperature dependent with the major portion of the NOx being converted to N_2 at all temperatures and the remaining fraction as N_2O. Without a Pt catalyst, 20% of the NOx is transformed to a species that is not detected by the CLA or FTIR.

In actual diesel exhaust the NOx reduction activity of the system is less that that measured for simulated exhaust. Using propylene injection to bring the C_1 to NOx ratio to 6, the system reduces 45-50% of the NOx. Without propylene injection the C_1:NOx out of the engine is 0.4:1 and the system does not significantly reduce NOx. After two days of testing some reduction in activity was observed. Tests are ongoing to determine why performance in simulated exhaust exceeds that in actual diesel exhaust and to determine if any permanent catalyst degradation occurred during engine testing.

ACKNOWLEDGMENTS

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CONTACT

Dr. M. Lou Balmer
Pacific Northwest National Laboratory
P.O. Box 999, MSIN K8-93
Richland, WA 99352
Lou.balmer@pnl.gov