ENRICHED AIR INSIDE A MUFFLE FURNACE

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ABSTRACT

This manuscript describes laboratory tests and calculations that explore the effectiveness of a stream of ionized air to oxidize soot and, thus, regenerate diesel particulate filters. Soot was oxidized inside a muffle furnace in two different configurations, either as a layer of soot spread in a porcelain boat, or as a quantity of soot evenly loaded in ceramic wall-flow monolith. Oxidation took place in air, ozone-enriched air or air ionized by an electric arc (thermal plasma), at furnace temperatures in the range of 200-450° C. It was found that when ozone was generated in the inlet air (1060 ppm) the consumption rate of soot increased by up to ten percent. However at the presence of the thermal plasma (generating O, NO_2, NO, and O_3) the carbon consumption was accelerated by factors varying from a few percent to often exceeding one hundred percent. The effectiveness of this technique depended on the characteristics of the arc. Moreover, the oxidation rate increased with increasing furnace temperature. Results also showed that the oxidation of diesel soot was much faster than that of pure carbon black and that the effect of the generated oxidizing species (O, O_3 NO_2, etc.) was more pronounced in the case of diesel soot. The present ozone generator consumed 10 W of electric power and the electric arc transformer was rated at 220 W. So far the results of this work suggest that the effect of ozone in oxidizing soot is minor, in this temperature range, while the effect of other oxidizing species (O, NO_2) could be significant. In vehicle applications the effluent of a particulate trap regenerated with this technique must be scrutinized, as it may contain toxic pollutants such as NO2. If the regeneration is performed off-line, the effluent of the trap may be channeled back to the engine, where further reactions may minimize the pollutants.

Key words: muffle, oxidize, enriched, pollutant, ceramic.

INTRODUCTION

Progress has been made by installing electronic engine controls in new generations of diesel engines, see for instance the common-rail injection pump or by mounting flow-through catalysts at the tail pipe of the vehicle. However, the results from such methods are deemed inadequate to satisfy stringent near-future regulations (Euro III and US2004) to ensure a clean environment. Diesel engines are emitters of particulates (mostly soot) and oxides of nitrogen (NO_x). The emissions problem becomes more exacerbated for old diesel engines, the life span of which can easily exceed 20 years. Unfortunately, it is quite common to see diesel vehicles emit-ting thick plumes of smoke in the center of towns around the word. Particulate emissions can be drastically reduced by installing after treatment devices at the exhaust of the engine. Such devices employ filters of different structure and materials to retain soot.

Ceramic honeycomb filters have gathered special popularity because of their high filtration area to volume ratio, good particle collection efficiency and good strength and durability at typical exhaust manifold temperatures of 200-500° C. However, severe problems have been encountered with the periodic regeneration (cleaning) of these filters and, over the past 15-20 years a great deal of research has been conducted in this topic. The most typical method for regeneration has been thermal regeneration, where the filter is heated to a sufficiently high temperature for the oxidation of car-bon to proceed at a high rate. While periodic thermal regeneration of these filters could be effective it often involved complicated hardware and caused thermal failures by cracking and melting of the filters.

Work at Northeastern University, over the last ten years, has concentrated in an alternative method, i.e., aerody-namic regeneration. In this technique, short bursts of compressed air (tank pressure of 4-6 atm and inlet filter pressure of 1-2 atm, gage) are used to dislodge the soot from the filter and channel it to a collection system, which may include a burner as an option. This method is used optimally with surface filters, such as wall-flow monoliths. Since the operating temperature of such ceramic monoliths can always be kept low (150 – 300° C), failures of the ceramic are prevented. There-fore, the durability and reliability of the trap is ensured. High filtration efficiency monoliths have been tested and shown to remove as much as 99\% of the total mass of diesel particulates. Combination of such highly effective monoliths with filtered exhaust gas recirculation (EGR) have been proven to drastically reduce the NO_x emissions as well.

This investigation explored an alternative method for the regeneration of diesel particulate filters. The collected soot is oxidized in the filter using *thermal regeneration*, but at the relatively low temperatures normally found in the diesel exhaust (200-500° C). Low temperatures can be afforded by using oxidation enhancement agents (O, O_3, NO_2, etc.). Using a reasonably small concentration of such oxidation agents in air, the necessary temperature for expedient oxidation of soot can be lowered. Thus, the advantage of using gas-phase oxidation agents becomes obvious; heating the filter to under 350-400° C, instead of 550-600° C [26], minimizes thermal stresses and `hot spots' and, hence, the likelihood of failure. Operating temperatures in the vicinity of 350° C may be similar to those that would have been employed in a catalytic trap oxidizer. However, the cases where the catalyst is applied to the filter itself are likely to experience ineffec-tive contact of the soot with the catalyzed filter surface, poor combustion control and high local reaction rates and, eventually, degradation of the coating. Moreover, the cases where the catalyst is in the form of a fuel additive, again experience inefficiencies of contacting the soot particles and possible emission of toxic metal com-pounds. Using a gas phase reaction agent, such as atomic oxygen, ozone, etc., to enhance the oxidation chemistry of soot and allow trap operation at lower tem-peratures appears to be a preferred method.

This paper reports on preliminary experiments, con-ducted in the laboratory, to assess the effectiveness of ($O/O_3/NO_2$/etc.)-enriched air to accelerate the oxidation of carbon. Experiments were conducted in an electrically-heated laboratory furnace where predetermined quanti-ties of carbonaceous particulates were burned at preset temperatures and gas flow rates. The oxidation rate was deduced by measuring the weight change of the particu-lates at set time intervals.

EXPERIMENTAL PROCEDURE

Experiments were conducted in the laboratory, where carbon was oxidized at steady state - steady laminar flow conditions, in an electrically-heated muffle furnace. Two types of carbon were oxidized: soot from the effluent of a diesel engine and pure carbon lamp black (purchased from *Fisher Scientific*, with granular size 44 nm. The diesel soot was collected from a particulate trap at the exhaust of a 1980 *Volkswagen Rabbit* diesel vehicle. It represents a typical mixture of soot produced under many different conditions, including city and highway driving under a variety of engine loads and speeds. Because of its residence period in the particulate trap the soot had a low volatile content (several percent). Typical road test conditions are depicted in the data shown. Both carbon samples were pre-dried in an oven at 90° C for 24 hours. The furnace was preheated at temperatures in the range of 200-450° C and, subsequently, the carbon sample was inserted in a 2 cm i.d. *Pyrex* tube placed in the furnace. Two methods were implemented for holding the soot in the test setup. In the more ‘traditional’ method a thin layer of soot (0.1 or 0.2 g) was evenly-spread in a porcelain boat. Even at the low temperatures of this study it was expected, based on the analysis shown in the Appendix, that bed diffusion could have been a limiting factor in the oxidation of carbonaceous particles in the porcelain boat experiments, at the presence of an oxidation enhancement agent. This was the case for the runs at temperatures of 350-450° C.

Thus, to minimize intra particle diffusion limitations in the bed of carbonaceous particulates, additional experiments were performed where the particulates were loaded in a cylindrical ceramic filter, which was inserted in the *Pyrex* tube and was placed at the mid-length distance of the furnace. The 1.7 cm diameter, 5 cm long section of the ceramic filter was cut from a cordierite wall flow monolith (200 channels/in^2), manufactured by *NGK*. The filter was loaded with particulates by connecting one of its ends to a vacuum pump and slowly passing the other end over a bed of soot. Particulates were, thus, sucked into the filter. The filter was then wrapped in high-temperature ceramic blanket (*Zircar*) and placed in the furnace tube. These lat-ter experiments not only provided better contact of the particulates in the filter with the oxidizer gas by minimiz-ing diffusion limitations but, also, approximated more closely the actual trap oxidizer systems. Schematics of the experimental setups are shown in Fig. 1. In the experiments where the thermal plasma was used, the ionized gas was generated at the entrance of the furnace *Pyrex* glass tube. The maximum power rating of this trans-former for generating the arc, between two electrodes, was 220 W (10000 Volts, 22 mA). In the other set of experiments an ozone generator (*AZCO Industries, Model HTU500-DC*) was used, powered by a 12VDC/ 800mA (9.6 W)supply. This generator was fed an 0.5 lpm oxygen stream and it produced 5,100 ppm of ozone. Upon blending the oxygen stream with nitrogen to pro-duce a 0.21/0.79 mixture ratio, approximating air, the ozone partial pressure in the furnace was reduced to a calculated value of 1062 ppm.

RESULTS AND DISCUSSION

Tests were initially conducted at room temperature to assess the repeatability of the technique. Samples of soot were placed in the porcelain boat, were inserted in the furnace and were left therein for preset periods of time, using the flowrates of air that were actually used in the elevated temperature tests. Upon the termination of such tests, the samples experienced minimal weight losses (up to 0.5%), due to handling. Heating samples of soot to 350° C for 10 min in nitrogen resulted in 9% losses, which are attributed to volatile gasification. All samples were cooled in the furnace in a flow of nitrogen, upon termination of each test, before being weighed.

Combustion of soot in ozone-enriched air (with the corona-discharge ozone generator).Tests in ozone-enriched air (1060 ppm of O_3) were conducted at 300, 350 and 400° with fixed beds of soot (□ 0.15 g). All tests were repeated in air. Results are shown in Fig. 2, expressed as \% of mass loss. It appears that the presence of ozone enhances the oxidation of soot, but the effect is rather mild (<10%) As an example, at 350° C 20% of carbon was gasified in 10 minutes in air (after the release of *approx* 9-10\% of volatiles), while 21.5% were gasified in ozone-enriched air. Under the same conditions, 35\% of soot was gasified in ionized air with the electric arc. These experiments are described in the next section.

Combustion of soot in ionized air (at the presence of the thermal plasma). Most data was collected in experiments where a thin layer of carbon particulates, of a fixed sample weight of 0.1 or 0.15 g, was placed in porcelain boats and was oxidized at fur-nace temperatures in the range of 200 – 450° C. Beds of either carbon black or diesel soot were oxidized in these experiments. These particulates were not pre-pyrolyzed. The profile of an experimental run. Several trends are apparent from the data.

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