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The preparation of this report is based on work funded in part by the State of Texas through a Grant from the Texas Commission on Environmental Quality.
Task 2 Report – Adsorption and Filtration Efficiencies

For the
TCEQ NTRD Project

Low-Cost Control of NOx, VOC and Soot from Stationary Diesel Generators Employing an Adsorptive Ceramic Filter with Microwave Regeneration

NTRD Project No. 582-5-70807-0007

Prepared for:
Texas Commission on Environmental Quality
12118 Park 35 Circle
Austin, Texas 78753

Prepared by:
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April 30, 2006
Executive Summary

This report is prepared and submitted to TCEQ to fulfill the Task 2 deliverables as stated in Section 2.2.3. of this NTRD (New Technology for Research and Development) project. The Task 2 of the project is described below.

Task 2. THE EXPERIMENTAL EVALUATION OF ADSORPTION EFFICIENCY OF NOX/VOC AND FILTRATION EFFICIENCY OF SOOT FOR THE CONSTRUCTED UNIT

2.2. Task Statement: The PERFORMING PARTY will conduct experiments to evaluate the efficiencies of adsorption and filtration on the constructed prototypes.

2.2.1. The PERFORMING PARTY will conduct experiments to evaluate the efficiencies of adsorption and filtration on the constructed prototypes.

2.2.1.1. The PERFORMING PARTY will connect exhausts from diesel generators to the constructed units to evaluate adsorption and filtration efficiencies.

2.2.1.2. The PERFORMING PARTY will analyze the upstream and downstream concentrations of NOx, VOC and soot to evaluate the sorption efficiency.

2.2.1.3. The PERFORMING PARTY will measure the NOx using a NOx analyzer; the VOC will be analyzed as total hydrocarbons using a total organic carbon analyzer and speciated using a GC/FID/MS; and the soot will be measured using two cascade impactors.

2.2.1.4. The PERFORMING PARTY will conduct these experiments with virgin and regenerated catalysts, and the effects of thermal aging and sulfur/ash poisoning will be investigated.

2.2.2. Schedule: The PERFORMING PARTY shall complete this task within 11 month of the signed Notice to Proceed Date as issued by TCEQ.

2.2.3. Deliverables: The PERFORMING PARTY shall submit a technical report describing the test procedures and test results.

Soot Filtration and NOx Reduction Efficiencies

The constructed diesel emission control prototype unit and the diesel emission test facilities shown in Figures 1 through 6 have been used in carrying out the Task 2 experiments. The experiments are designed to evaluate the soot filtration and the NOx reduction efficiencies. While additional experiments are currently still being carried out, this report summarizes the
results observed up to this stage. The experimental procedures and the typical results observed for the two investigations, namely, soot filtration efficiency and NOx reduction efficiency, associated with the constructed prototype emission control unit shown in Figure 1 are reported separately in the following two sections.

A. Soot Filtration

After the instrument calibration and a couple of shake-down runs, the constructed prototype unit was installed in the system with soot samples before and after the DPF being collected in the sampler. The collected soot samples were weighed by a high-precision microwave balance to determine the filtration efficiency. It was generally observed that a high soot removal efficiency of better than 90% was obtained based on a 10 minute-sampling time. The exhaust gas was also analyzed for O2, NO, NO2, CO and SO2 by the Testo-350 Gas Analyzer. A typical set of exhaust gas conditions is given in Table 1 below for reference.

<table>
<thead>
<tr>
<th>Exhaust Load (m³/hr)</th>
<th>Temperature (°C)</th>
<th>Press *H₂O (ppm)</th>
<th>DP *H₂O (ppm)</th>
<th>Outlet O₂ (ppm)</th>
<th>Outlet NO (ppm)</th>
<th>Outlet NO₂ (ppm)</th>
<th>Outlet CO (ppm)</th>
<th>Outlet SO₂ (ppm)</th>
<th>Outlet CO₂ (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>50</td>
<td>36.4</td>
<td>400</td>
<td>248</td>
<td>48.77</td>
<td>38.88</td>
<td>8.51</td>
<td>607</td>
<td>22.9</td>
<td>650</td>
</tr>
</tbody>
</table>

Note: 1. Soot samples were taken after 1 hour warm up at 70% of electric load.
2. CO₂ concentration in %, and others in ppm.

B. NOx Reduction

Numerous series of NOx reduction tests have been carried out in the microwave test unit using the constructed prototype control unit shown in Figure 1 with both virgin and regenerated catalysts. It has been generally observed that the NOx (NO + NO₂) in the diesel exhaust is successfully reduced with the injection of hexane as a reducing agent. It is worth pointing out again that the designed catalyst pack includes an activated platinum gauze (wire mesh), a SiC foam discs (10 mm dia x 4 mm thick, 80 ppi), and followed by a VOC oxidation catalyst (honeycomb). Both the virgin and the regenerated catalysts have shown similar efficiencies. A typical set of experimental data is provided in Table 2 below for reference.

<table>
<thead>
<tr>
<th>Microwave Analysis</th>
<th>Engine Exhaust</th>
<th>Exhaust with DeNOx/VOC Catalyst</th>
</tr>
</thead>
<tbody>
<tr>
<td>Watt (Watt)</td>
<td>9.1</td>
<td>330</td>
</tr>
<tr>
<td>Bed Temp (°C)</td>
<td>538</td>
<td>1000</td>
</tr>
<tr>
<td>O₂ (%)</td>
<td>537</td>
<td>0.2</td>
</tr>
<tr>
<td>CO (ppm)</td>
<td>99</td>
<td>9</td>
</tr>
<tr>
<td>NO (ppm)</td>
<td>636</td>
<td>0</td>
</tr>
<tr>
<td>NO₂ (ppm)</td>
<td></td>
<td>1</td>
</tr>
<tr>
<td>NOx (ppm)</td>
<td></td>
<td>1</td>
</tr>
</tbody>
</table>

Note: Analysis done by Testo 350 Analyzer
Concluding Remarks

The constructed diesel emission control prototype unit has demonstrated high efficiencies on both soot filtration and NOx reduction capabilities. While the effects of sulfur/ash poisoning and thermal aging have not yet been observed from our current experiments, these effects are expected and will be characterized with additional experiments planned for the project during the next four months.
Figure 1.

The Constructed Diesel Emission Control Prototype Unit.
Figure 2.

Schematic Diagram of the Designed Holder for the Constructed Prototype.
Figure 3.

Figure 4.
Photo Picture of the Diesel Emission Test Facility.
Figure 5.

Diesel Emission Test with the Constructed Prototype Enclosed in a Microwave Oven.
Figure 6.

The Waveguide Design for the Constructed Prototype.
Task 3 Report – Microwave Regeneration and NOx/VOC/Soot Destruction Efficiencies

For the TCEQ NTRD Project

Low-Cost Control of NOx, VOC and Soot from Stationary Diesel Generators Employing an Adsorptive Ceramic Filter with Microwave Regeneration

NTRD Project No. 582-5-70807-0007

Prepared for:
Texas Commission on Environmental Quality
12118 Park 35 Circle
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Prepared by:
Thomas C. Ho, PI
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P. O. Box 10053
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April 30, 2006
Executive Summary

This report is prepared and submitted to TCEQ to fulfill the Task 3 deliverables as stated in Section 2.3.3. of this NTRD (New Technology for Research and Development) project. The Task 3 of the project is described below.

Task 3. **THE CORRESPONDING EVALUATION OF DESTRUCTION EFFICIENCY OF NOX/VOC AND SOOT UPON MICROWAVE IRRADIATION IN THE REGENERATION PROCESS**

2.3. Task Statement: The PERFORMING PARTY will conduct experiments to evaluate the destruction efficiencies of NOx, VOC and soot upon microwave regeneration.

2.3.1. The PERFORMING PARTY will conduct experiments to evaluate the destruction efficiencies of NOx, VOC and soot upon microwave regeneration.

2.3.1.1. The PERFORMING PARTY will conduct these experiments simultaneously with those of Task 2.

2.3.1.2. The PERFORMING PARTY will focus these experiments on the destruction efficiencies upon microwave irradiation.

2.3.1.3. The PERFORMING PARTY will analyze the upstream and downstream NOx, VOC and soot to evaluate the efficiencies.

2.3.1.4. The PERFORMING PARTY will conduct these experiments with virgin and regenerated catalysts.

2.3.2. Schedule: The PERFORMING PARTY shall complete this task within 11 month of the signed Notice to Proceed Date as issued by TCEQ.

2.3.3. Deliverables: The PERFORMING PARTY shall submit a technical report describing the test procedures and test results to the TCEQ.

Microwave Regeneration and NOx/VOC/Soot Destruction Efficiencies

The constructed diesel emission control prototype unit and the diesel emission test facilities shown in Figures 1 through 6 have been used in carrying out the Task 3 experiments. The experiments are designed to evaluate the efficiencies of microwave regeneration and destruction of NOx/VOC/Soot upon microwave irradiation. While additional experiments are currently still being carried out, this report summarizes the results observed up to this stage. The experimental procedures and the typical results observed for the two investigations, namely, microwave regeneration efficiency and NOx/VOC/Soot destruction efficiency, associated with the constructed prototype emission control unit shown in Figure 1 are reported separately in the following two sections.
A. Microwave Regeneration

Microwave regeneration of the constructed diesel emission control prototype unit involves the use of microwave energy to oxide the filtered solid particulates (soot) to gaseous products (CO and CO$_2$) at elevated temperatures of 500-600 °C. Since silicone carbide (SiC) exhibits high RF absorption properties (high dielectric constant), it can be effectively heated by microwave. In addition, since the soot has good microwave absorption properties, the heating of soot by the microwave energy to induce combustion is effective as well. In our experiments, the removal of soot has been performed either continuously during regular operation of the prototype unit (on-line regeneration), or periodically after a pre-determined quantity of soot has been accumulated (off-line regeneration). Numerous regeneration tests have been carried out using the constructed prototype unit. During the tests, the temperature of the filter is monitored by a temperature sensor located in the center of the filter. Pressure drop across the filter, CO concentration and filter temperature are simultaneously recorded in the data acquisition system. Typical results are shown in the following sections.

Radial Temperature Profiles

Figure 7 displays typical radial temperature profiles in the constructed prototype unit. As indicated, the radial temperature profiles are relatively uniform within about 100°F during microwave regeneration.

Vertical Temperature Profiles

Figure 8 displays typical vertical temperature profiles in the constructed prototype unit. Again, the vertical temperature profiles are also uniform within about 100°F during microwave regeneration.

Pressure Drop during Regeneration

Figure 9 displays typical pressure drop data during microwave regeneration for a plugged and an unplugged unit. The results indicated the regeneration is successfully completed at the end of the process.

Temperature vs CO Plot

Figure 10 shows a typical plot of temperature vs CO Concentration during Microwave Regeneration. It indicates that the CO concentration jumps up at the temperature of 300°C indicating the start of the soot combustion process.

Temperature vs Pressure Drop Plot

Figure 11 is a typical plot of temperature vs pressure drop during Microwave Regeneration. As indicated, the pressure drop starts to decrease at a temperature of 400°C and eventually returns to the pressure drop corresponding to a clean unit, which indicates complete regeneration.
Optimum Regeneration Duration

The regeneration tests conducted so far have indicated that majority of the soot is burnt off in the first 5 minutes and at > 400 ºC, and while maintaining DPF temperature at 600-650ºC for remaining 15 minutes, slight changes in the filter DP (differential pressure) and CO concentration have been observed. These tests suggest an optimum regeneration time of 10 minutes with air.

B. NOx/VOC/Soot Destruction

The results shown above have demonstrated the soot destruction capability of the proposed microwave regeneration technology. As reported in the Task 2 Report, numerous series of NOx/VOC destruction tests have been carried out in the microwave test unit using the constructed prototype control unit shown in Figure 1 with both virgin and regenerated catalysts. It has been generally observed that the NOx (NO + NO2) in the diesel exhaust is successfully reduced with the injection of hexane as a reducing agent. Both the virgin and the regenerated catalysts have shown similar efficiencies. The VOC destruction efficiency is expected to be high but has not been confirmed due to the limitation of the Testo 350 Analyzer. Additional analysis method will be used to characterize

Concluding Remarks

The constructed diesel emission control prototype unit has demonstrated high efficiencies on soot filtration, soot regeneration and NOx destruction capabilities. While the effects of sulfur/ash poisoning and thermal aging have not yet been observed from our current experiments, these effects are expected and will be characterized with additional experiments planned for the project during the next four months.
Figure 1.

The Constructed Diesel Emission Control Prototype Unit.
Figure 2.

Schematic Diagram of the Designed Holder for the Constructed Prototype.
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Figure 4.

Photo Picture of the Diesel Emission Test Facility.
Figure 5.

Diesel Emission Test with the Constructed Prototype Enclosed in a Microwave Oven.
Figure 6.

The Waveguide Design for the Constructed Prototype.
Figure 7.

Typical Radial Temperature Profile in the Prototype Unit.
Figure 8.

Typical Vertical Temperature Profile in the Prototype Unit.
Figure 9.

Pressure Drop Profiles Associated with Plugged and Clean Prototype Units.
Figure 10.

Plot of Temperature vs CO Concentration during Microwave Regeneration.
Figure 11.

Plot of Temperature vs Pressure Drop during Microwave Regeneration.
Task 4 Report – The Investigation of Optimal Design and Operation Parameters

For the
TCEQ NTRD Project

Low-Cost Control of NOx, VOC and Soot from Stationary Diesel Generators Employing an Adsorptive Ceramic Filter with Microwave Regeneration

NTRD Project No. 582-5-70807-0007

Prepared for:
Texas Commission on Environmental Quality
12118 Park 35 Circle
Austin, Texas 78753

Prepared by:
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November 30, 2006
Executive Summary

This report is prepared and submitted to TCEQ to fulfill the Task 4 deliverables as stated in Section 2.4.3. of this NTRD (New Technology for Research and Development) project. The Task 4 of the project is described below.

Task 4. THE INVESTIGATION OF OPTIMAL DESIGN AND OPERATION PARAMETERS

2.4. Task Statement: The PERFORMING PARTY will investigate the optimal operating parameters leading to maximum NOx, VOC, and soot control.

2.4.1. The PERFORMING PARTY will examine experimental and operating conditions effects on control efficiencies. These may include microwave energy level, porosity and thickness of ceramic filter, catalyst pre-treatment, catalyst size, catalyst amount, packing pattern and etc. The pressure drop across the device under various operating conditions will be carefully characterized.

2.4.2. Schedule: The PERFORMING PARTY shall complete this task within 18 month of the signed Notice to Proceed Date as issued by TCEQ.

2.4.3. Deliverables: The PERFORMING PARTY shall submit a final technical report describing the optimal operating conditions of the constructed prototypes to the TCEQ.

Review of Task 3 Results - Microwave Regeneration and NOx/VOC/Soot Destruction Efficiencies

As described in the submitted Task 3 Report, the constructed diesel emission control prototype unit and the diesel emission test facilities shown in Figures 1 through 6 have been used in carrying out the Task 3 experiments. The experiments are designed to evaluate the efficiencies of microwave regeneration and destruction of NOx/VOC/Soot upon microwave irradiation. The experimental procedures and typical results observed for the two investigations, namely, microwave regeneration efficiency and NOx/VOC/Soot destruction efficiency, associated with the constructed prototype emission control unit shown in Figure 1 are reported separately in the two sections below.

A. Microwave Regeneration

Microwave regeneration of the constructed diesel emission control prototype unit involves the use of microwave energy to oxide the filtered solid particulates (soot) to gaseous products (CO and CO2) at elevated temperatures of 500-600 °C. Since silicone carbide (SiC) exhibits high RF absorption properties (high dielectric constant), it can be effectively heated by microwave. In addition, since the soot has good microwave absorption properties, the heating of soot by the microwave energy to induce combustion is effective as well. In our experiments, the removal of
soot has been performed either continuously during regular operation of the prototype unit (on-line regeneration), or periodically after a pre-determined quantity of soot has been accumulated (off-line regeneration). Numerous regeneration tests have been carried out using the constructed prototype unit. During the tests, the temperature of the filter is monitored by a temperature sensor located in the center of the filter. Pressure drop across the filter, CO concentration and filter temperature are simultaneously recorded in the data acquisition system. Typical results are shown in the following sections.

Radial Temperature Profiles

Figure 7 displays typical radial temperature profiles in the constructed prototype unit. As indicated, the radial temperature profiles are relatively uniform within about 100°F during microwave regeneration.

Vertical Temperature Profiles

Figure 8 displays typical vertical temperature profiles in the constructed prototype unit. Again, the vertical temperature profiles are also uniform within about 100°F during microwave regeneration.

Pressure Drop during Regeneration

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Figure 11 is a typical plot of temperature vs pressure drop during Microwave Regeneration. As indicated, the pressure drop starts to decrease at a temperature of 400°C and eventually returns to the pressure drop corresponding to a clean unit, which indicates complete regeneration.

Optimum Regeneration Duration

The regeneration tests conducted so far have indicated that majority of the soot is burnt off in the first 5 minutes and at > 400 °C, and while maintaining DPF temperature at 600-650°C for remaining 15 minutes, slight changes in the filter DP (differential pressure) and CO concentration have been observed. These tests suggest an optimum regeneration time of 10 minutes with air.
B. NOx/VOC/Soot Destruction

The results shown above have demonstrated the soot destruction capability of the proposed microwave regeneration technology. As reported in the Task 2 Report, numerous series of NOx/VOC destruction tests have been carried out in the microwave test unit using the constructed prototype control unit shown in Figure 1 with both virgin and regenerated catalysts. It has been generally observed that the NOx (NO + NO2) in the diesel exhaust is successfully reduced with the injection of hexane as a reducing agent. Both the virgin and the regenerated catalysts have shown similar efficiencies. The VOC destruction efficiency is expected to be high but has not been confirmed due to the limitation of the Testo 350 Analyzer.

Summary of Task 4 Results - The Investigation of Optimal Design and Operation Parameters

The constructed diesel emission control prototype unit and the diesel emission test facilities shown in Figures 1 through 6 have continuously been used in carrying out the Task 4 experiments. These experiments are designed to evaluate the optimal design and operation parameters associated with the developed technology. The experimental procedures and typical results observed are summarized separately in the following two sections.

A. Optimal Parameters for DPF Regeneration

As described previously, microwave regeneration of the constructed diesel emission control prototype unit involves the use of microwave energy to oxide the filtered solid particulates (soot) to gaseous products (CO and CO2) at elevated temperatures of 500-600 °C. Since silicone carbide (SiC) exhibits high RF absorption properties (high dielectric constant), it can be effectively heated by microwave. In addition, since the soot has good microwave absorption properties, the heating of soot by the microwave energy to induce combustion is effective as well. In our experiments, the removal of soot has been performed either continuously during regular operation of the prototype unit (on-line regeneration), or periodically after a predetermined quantity of soot has been accumulated (off-line regeneration). Numerous regeneration tests have been carried out using the constructed prototype unit. During the tests, the temperature of the filter is monitored by a temperature sensor located in the center of the filter. Pressure drop across the filter, CO concentration and filter temperature are simultaneously recorded in the data acquisition system. Summarized results are reported below.

Soot Loading Rate

To effectively investigate optimal parameters for DPFR regeneration, a new control valve was installed in the DPF regeneration facility for maintaining a constant exhaust gas flow rate during the regeneration tests. The modification enabled the project to accurately estimate the soot loading rate through measuring the differential pressure drop of DPF at a particulate flow rate. A series of tests were performed and a typical set of correlation results between the soot loading rate and the differential pressure drop reading is given in Figure 12. As indicated in Figure 12, the soot loading rate is seen to be proportional to the measured differential pressure drop across
the DPF. The observed correlation allows the project to estimate the amount of soot loading on the DPF based on the measured differential pressure drop.

**Microwave DPF Regeneration**

Numerous sets of DPF regeneration tests were conducted to investigate the DPF regeneration characteristics involving microwave heating. Experimental observations from three typical regeneration tests (5 cycles for each test) are displayed in Figures 13-15, where Figure 13 is for Test 6-163 with a 10 minutes regeneration time and Figures 14 and 15 are for Test 6-165 with a 5 minutes regeneration time. The results shown in Figure 13 indicate that, once the engine was turned on, the differential pressure started to increase due to soot loading and the increase in temperature. This pressure reached to about 50 inch of H₂O in about 20 minutes. During the period, the exhaust gas temperature was also seen to increase from room temperature to about 250 °C. No soot combustion was observed at this temperature because it was below the soot ignition temperature. At this moment when the differential pressure reached 50 inch of H₂O, the microwave power was turned on and the exhaust gas temperature was seen to rise above the soot ignition temperature at about 400°C. This elevated temperature ignited the soot and the heat generated from soot combustion further elevates the gas temperature to its maximum of higher than 700°C. The differential pressure was seen to simultaneously return to near zero at the end of the regeneration. Similar observations were repeated for the next four regeneration cycles. It is essential to note that, after each regeneration cycle, the differential pressure always returns to near zero indicating high regeneration efficiencies. Similar regeneration trends were observed for Test 6-165 as shown in Figures 14 and 15. In these two figures, the CO concentration is also plotted, where the peaks of CO are observed to be synchronized with the temperature peaks. The observations are expected since both are associated with soot combustion.

It should be pointed out that the duration of microwave heating between 10 and 5 minutes does not generate any difference on the regeneration of DPF as indicated in Figures 13 and 15. Although not shown, this observation has been confirmed throughout the tests conducted during the Task 4 investigation. The main reason for this observation is that the microwave energy only serves to raise the exhaust gas temperature to reach the ignition temperature of the trapped soot to create soot combustion. The heat generated from soot combustion further raises the exhaust gas temperature to reach its maximum. The temperature then cools down as soon as the soot combustion is completed. It is worth mentioning that, once the soot combustion starts, the continuous supply of heat from microwave power becomes unnecessary since it is insignificant as compared to that generated from soot combustion. The use of 5 minutes of microwave heating, therefore, is sufficient and more energy efficient for microwave regeneration of the developed protocol.

It should also be reported that the original DPF filter was taken out from the holder for examination after 20 microwave regeneration tests including 15 off-line and 5 on-line. No apparent physical damages on filter cells were observed. The only noticeable difference was that a slight increase in pressure drop was observed as compared to a new filter. An accumulation of soot ash in the DPF wall was suspected to cause this slightly higher pressure drop. Although not shown, the off-line regeneration has been observed to be more efficient in DPF regeneration than on-line regeneration due to its higher oxygen content.
B. Optimal Parameters for NOx Control

As reported in the Task 2 Report, numerous series of NOx/VOC destruction tests have been carried out in the microwave test unit using the constructed prototype control unit shown in Figure 1 with both virgin and regenerated catalysts. It has been generally observed that the NOx (NO + NO2) in the diesel exhaust is successfully reduced with the injection of hexane as a reducing agent. Both the virgin and the regenerated catalysts have shown similar efficiencies. Additional tests have been conducted and the following sections summarize optimal operating conditions based on the results observed.

Optimal DENOX Catalyst Temperature

Many series of NOx reduction tests have been completed in the microwave test facility first using a NO standard of 1045 ppm of NO to characterize the effects of residence time, catalyst bed temperature and hexane injection rate on NO reduction. After many modifications, the current system has been able to achieve an excellent and consistent NO reduction result. A typical set of such results representing the current system are shown in Table 1, where the experiments were conducted under 900°C of bed temperature with three NO flow rates. The hexane injection rate was the same at 10 cm³/min for all the three NO flow rates. The results shown in the table indicate that the NO conversion efficiencies are extremely high at more than 99%. However, the CO oxidation is not as good with higher than 500 ppm still in the treated gas. This CO problem has later been solved as will be reported in the section below. It should be noted that, after numerous tests, it has been concluded that the optimal DENOX bed temperature for the developed catalyst is between 900°C to 1100°C.

In addition to employing NO standards in the NO reduction tests, real engine exhaust has also been involved in the tests. A typical set of such results are shown in Table 2, where the catalyst bed temperature is at 1000°C with four hexane injection rates at 0, 0.1., 0.15, and 0.2 of the engine exhaust. The results again indicate that the NOx reduction efficiencies are excellent at higher than 93% when hexane is injected. Without hexane injection, the efficiency is seen to drop down to about 78%. Among the three hexane injection rates, the one with 10% of engine exhaust appears to generate the best result with a 97.6% of NO reduction efficiency. Again, the results indicate that CO oxidation is not effective with the current system. It is worth reporting that the high temperature in the DENOX catalyst bed may cause a trace amount of SiC to react with oxygen to form CO. The generated CO, however, enhances NO reduction on the catalyst bed as can be seen in a typical set of results shown in Figure 16.

Optimal CO/VOC Catalytic Temperature

As reported in the previous section, the developed catalyst beds have been effective for NO reduction but not for CO oxidation. After many tests and modifications based on literature reports, it has finally been identified that the CO/VOC catalyst was not effective because it was not operated at the optimal temperature. In the previous design, the two catalyst beds, i.e., the DENOX and the CO/VOC beds, were line up in the developed protocol under the same microwave power, which created a higher than needed temperature for the CO/VOC catalyst bed. The CO/VOC bed has since been separated from the DENOX bed in the redesign of
microwave waveguide. The optimal temperature for the CO/VOC bed has then been tested to be around 200-400°C and the CO oxidation efficiency is seen to improve dramatically as can be seen from the results shown in Table 3. These results indicate that CO may be generated from the DENOX bed at 436 ppm and the generated CO has subsequently been oxidized in the CO/VOC bed and been decreased to 10 ppm. In addition, the CO/VOC catalyst is capable of reducing the NOx further from 296 ppm down to 3 ppm with the existence of CO. Similar results can be observed in the results shown in Figure 17. These results in the figure indicate that the microwave heating induces the formation of CO from SiC, the formed CO is subsequently decreased to near zero when the CO/VOC catalyst is made on-line, the CO reappears after the CO/VOC is made off-line, the CO level increases further once the hexane is injected, the CO again decreases to zero when the CO/VOC catalyst is made on-line again. While the CO concentration varies, the NO concentration remains low during the entire period when the microwave heating is on. Several additional plots showing typical results from DENOX tests are displayed in Figures 18-20. Figure 18 shows results from a typical de-NOx test involving a standard NO stream of 1021 ppm, while Figures 19 and 20 show typical results associated with de-NOx tests involving actual engine exhaust. As can be seen in the figures, the effectiveness of the using of hexane and catalyst beds in the reduction of NO in the exhaust stream is well-indicated. The optimal hexane flow rate has been determined to be at 2.5% of the engine exhaust flow rate.

Concluding Remarks

DPF regeneration employing microwave heating is effective and energy efficient. The required microwave heating is only to raise the exhaust gas temperature to the ignition temperature of the trapped soot at about 400°C. The heat generated from soot combustion will then complete the regeneration process. In the developed protocol, a 5 minute of microwave heating is sufficient to induce soot combustion for each regeneration cycle. The modified catalyst beds design in the protocol is seen to produce an effective and consistent control of both NOx and CO from diesel exhaust. The optimal bed temperatures for the two catalyst beds appear to be 900-1100°C for the DENOX bed and 200-400°C for the CO/VOC bed. The injection of hexane enhances the NO reduction efficiency while, even without hexane injection, the high temperature bed may promote the formation of CO from SiC, which results in the enhancing of NO reduction on the DENOX and CO/VOC catalysts. The optimal hexane flow rate has been determined to be at 2.5% of the engine exhaust flow rate.
Table 1. NOx Reduction Test Involving Standard NO Stream – Effect of NO Flow Rate

<table>
<thead>
<tr>
<th>NO Flow</th>
<th>Bed Temp (°C)</th>
<th>Analysis (Testo 350)</th>
<th>O2</th>
<th>CO</th>
<th>NO</th>
<th>NO2</th>
<th>NOx</th>
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<tr>
<td>Standard</td>
<td>0.3</td>
<td>0</td>
<td>1045</td>
<td>31</td>
<td>1076</td>
<td></td>
<td></td>
</tr>
<tr>
<td>DeNOx + VOC/CO catalyst</td>
<td>100</td>
<td>900</td>
<td>0.2</td>
<td>4800</td>
<td>11</td>
<td>0</td>
<td>11</td>
</tr>
<tr>
<td>DeNOx + VOC/CO catalyst</td>
<td>200</td>
<td>900</td>
<td>0.2</td>
<td>1114</td>
<td>20</td>
<td>1</td>
<td>21</td>
</tr>
<tr>
<td>DeNOx + VOC/CO catalyst</td>
<td>300</td>
<td>900</td>
<td>0.2</td>
<td>366</td>
<td>26</td>
<td>0</td>
<td>26</td>
</tr>
</tbody>
</table>

Note: Hexane vapor injection remained at a constant rate of 10 cc/min.
NOx Reduction Test Involving Engine Exhaust – Effect of Hexane Injection Rate

Table 2. Engine Exhaust DeNOx Test

<table>
<thead>
<tr>
<th>Exhaust Flow (cc/min)</th>
<th>HC (cc/min)</th>
<th>O2 ppm</th>
<th>CO ppm</th>
<th>NO ppm</th>
<th>NO2 ppm</th>
<th>NOx ppm</th>
<th>SO2 ppm</th>
</tr>
</thead>
<tbody>
<tr>
<td>Engine Exhaust</td>
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<td>1.7</td>
<td>-</td>
<td>52</td>
<td>0</td>
<td>52</td>
<td>-</td>
</tr>
</tbody>
</table>

Note: (1). HC is hexane vapor
(2) Temperature of DeNOx catalyst bed is ~1000 °C
(3) VOC/CO catalyst appears inactive compared with the previous test.
### Table 3. DeNOx and CO/VOC Catalyst

<table>
<thead>
<tr>
<th>Catalyst*</th>
<th>NO Std Flow</th>
<th>HC&lt;sup&gt;(1)&lt;/sup&gt;</th>
<th>O2</th>
<th>CO</th>
<th>NO</th>
<th>NO2</th>
<th>NOx</th>
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<tr>
<td>NO Standard</td>
<td>0.2</td>
<td>0</td>
<td>1029</td>
<td>92</td>
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<tr>
<td>DeNOX</td>
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<td>436</td>
<td>290</td>
<td>6</td>
<td>296</td>
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<tr>
<td>CO/VOC</td>
<td>200</td>
<td>0</td>
<td>0.3</td>
<td>10</td>
<td>3</td>
<td>0</td>
<td>3</td>
</tr>
</tbody>
</table>

Note:  
(1) CO/VOC catalyst pack temperature = 219°C  
(2) DeNOX pack temperature = 950°C  
(3) NO standard is 1023 ppm NO in nitrogen

* DeNOX – Exhaust Analysis in the Exit Stream from the DeNOX Catalyst Bed  
CO/VOC – Exhaust Analysis in the Exit Stream from the CO/VOC Catalyst Bed
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Task 5 Report – The Development of a Commercialization Plan for the Technology

For the
TCEQ NTRD Project

Low-Cost Control of NOx, VOC and Soot from Stationary Diesel Generators Employing an Adsorptive Ceramic Filter with Microwave Regeneration

NTRD Project No. 582-5-70807-0007

Prepared for:
Texas Commission on Environmental Quality
12118 Park 35 Circle
Austin, Texas 78753

Prepared by:
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Lamar University
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November 30, 2006
Executive Summary

This report is prepared and submitted to TCEQ to fulfill the Task 5 deliverables as stated in Section 2.5.3. of this NTRD (New Technology for Research and Development) project. The Task 5 of the project is described below.

Task 5. THE DEVELOPMENT OF A COMMERCIALIZATION PLAN FOR THE TECHNOLOGY

2.5. Task Statement: The PERFORMING PARTY will develop a commercialization plan for the demonstrated technology.

2.5.1. The PERFORMING PARTY will develop a commercialization plan for the demonstrated technology.

2.5.1.1. The PERFORMING PARTY will consider the mass production of the device and the marking of the device in an economically sustainable manner in the commercialization plan.

2.5.1.2. The PERFORMING PARTY will seek advice from Lamar University’s Small Business Development Center in accomplishing the task.

2.5.2. Schedule: The PERFORMING PARTY shall complete this task within 18 month of the signed Notice to Proceed Date as issued by TCEQ.

2.5.3. Deliverables: The PERFORMING PARTY shall submit a commercialization plan to the TCEQ.

Review of Task 3 Results - Microwave Regeneration and NOx/VOC/Soot Destruction Efficiencies

As described in the submitted Task 3 Report, the constructed diesel emission control prototype unit and the diesel emission test facilities shown in Figures 1 through 6 have been used in carrying out the Task 3 experiments. The experiments are designed to evaluate the efficiencies of microwave regeneration and destruction of NOx/VOC/Soot upon microwave irradiation. The experimental procedures and typical results observed for the two investigations, namely, microwave regeneration efficiency and NOx/VOC/Soot destruction efficiency, associated with the constructed prototype emission control unit shown in Figure 1 are reported separately in the two sections below.

A. Microwave Regeneration

Microwave regeneration of the constructed diesel emission control prototype unit involves the use of microwave energy to oxide the filtered solid particulates (soot) to gaseous products (CO
and CO₂) at elevated temperatures of 500-600 °C. Since silicone carbide (SiC) exhibits high RF absorption properties (high dielectric constant), it can be effectively heated by microwave. In addition, since the soot has good microwave absorption properties, the heating of soot by the microwave energy to induce combustion is effective as well. In our experiments, the removal of soot has been performed either continuously during regular operation of the prototype unit (on-line regeneration), or periodically after a pre-determined quantity of soot has been accumulated (off-line regeneration). Numerous regeneration tests have been carried out using the constructed prototype unit. During the tests, the temperature of the filter is monitored by a temperature sensor located in the center of the filter. Pressure drop across the filter, CO concentration and filter temperature are simultaneously recorded in the data acquisition system. Typical results are shown in the following sections.

Radial Temperature Profiles

Figure 7 displays typical radial temperature profiles in the constructed prototype unit. As indicated, the radial temperature profiles are relatively uniform within about 100°F during microwave regeneration.

Vertical Temperature Profiles

Figure 8 displays typical vertical temperature profiles in the constructed prototype unit. Again, the vertical temperature profiles are also uniform within about 100°F during microwave regeneration.

Pressure Drop during Regeneration

Figure 9 displays typical pressure drop data during microwave regeneration for a plugged and an unplugged unit. The results indicated the regeneration is successfully completed at the end of the process.

Temperature vs CO Plot

Figure 10 shows a typical plot of temperature vs CO Concentration during Microwave Regeneration. It indicates that the CO concentration jumps up at the temperature of 300°C indicating the start of the soot combustion process.

Temperature vs Pressure Drop Plot

Figure 11 is a typical plot of temperature vs pressure drop during Microwave Regeneration. As indicated, the pressure drop starts to decrease at a temperature of 400°C and eventually returns to the pressure drop corresponding to a clean unit, which indicates complete regeneration.

Optimum Regeneration Duration

The regeneration tests conducted so far have indicated that majority of the soot is burnt off in the first 5 minutes and at > 400 °C, and while maintaining DPF temperature at 600-650°C for
remaining 15 minutes, slight changes in the filter DP (differential pressure) and CO concentration have been observed. These tests suggest an optimum regeneration time of 10 minutes with air.

**B. NOx/VOC/Soot Destruction**

The results shown above have demonstrated the soot destruction capability of the proposed microwave regeneration technology. As reported in the Task 2 Report, numerous series of NOx/VOC destruction tests have been carried out in the microwave test unit using the constructed prototype control unit shown in Figure 1 with both virgin and regenerated catalysts. It has been generally observed that the NO\textsubscript{x} (NO + NO\textsubscript{2}) in the diesel exhaust is successfully reduced with the injection of hexane as a reducing agent. Both the virgin and the regenerated catalysts have shown similar efficiencies. The VOC destruction efficiency is expected to be high but has not been confirmed due to the limitation of the Testo 350 Analyzer.

**Review of Task 4 Results - The Investigation of Optimal Design and Operation Parameters**

As described in the submitted Task 4 Report, the constructed diesel emission control prototype unit and the diesel emission test facilities shown in Figures 1 through 6 have continuously been used in carrying out the Task 4 experiments. These experiments are designed to evaluate the optimal design and operation parameters associated with the developed technology. The experimental procedures and typical results observed are summarized separately in the following two sections.

**A. Optimal Parameters for DPF Regeneration**

As described previously, microwave regeneration of the constructed diesel emission control prototype unit involves the use of microwave energy to oxide the filtered solid particulates (soot) to gaseous products (CO and CO\textsubscript{2}) at elevated temperatures of 500-600 °C. Since silicone carbide (SiC) exhibits high RF absorption properties (high dielectric constant), it can be effectively heated by microwave. In addition, since the soot has good microwave absorption properties, the heating of soot by the microwave energy to induce combustion is effective as well. In our experiments, the removal of soot has been performed either continuously during regular operation of the prototype unit (on-line regeneration), or periodically after a pre-determined quantity of soot has been accumulated (off-line regeneration). Numerous regeneration tests have been carried out using the constructed prototype unit. During the tests, the temperature of the filter is monitored by a temperature sensor located in the center of the filter. Pressure drop across the filter, CO concentration and filter temperature are simultaneously recorded in the data acquisition system. Summarized results are reported below.

**Soot Loading Rate**

To effectively investigate optimal parameters for DPFR regeneration, a new control valve was installed in the DPF regeneration facility for maintaining a constant exhaust gas flow rate during
the regeneration tests. The modification enabled the project to accurately estimate the soot loading rate through measuring the differential pressure drop of DPF at a particulate flow rate. A series of tests were performed and a typical set of correlation results between the soot loading rate and the differential pressure drop reading is given in Figure 12. As indicated in Figure 12, the soot loading rate is seen to be proportional to the measured differential pressure drop across the DPF. The observed correlation allows the project to estimate the amount of soot loading on the DPF based on the measured differential pressure drop.

Microwave DPF Regeneration

Numerous sets of DPF regeneration tests were conducted to investigate the DPF regeneration characteristics involving microwave heating. Experimental observations from three typical regeneration tests (5 cycles for each test) are displayed in Figures 13-15, where Figure 13 is for Test 6-163 with a 10 minutes regeneration time and Figures 14 and 15 are for Test 6-165 with a 5 minutes regeneration time. The results shown in Figure 13 indicate that, once the engine was turned on, the differential pressure started to increase due to soot loading and the increase in temperature. This pressure reached to about 50 inch of H$_2$O in about 20 minutes. During the period, the exhaust gas temperature was also seen to increase from room temperature to about 250 °C. No soot combustion was observed at this temperature because it was below the soot ignition temperature. At this moment when the differential pressure reached 50 inch of H$_2$O, the microwave power was turned on and the exhaust gas temperature was seen to rise above the soot ignition temperature at about 400°C. This elevated temperature ignited the soot and the heat generated from soot combustion further elevates the gas temperature to its maximum of higher than 700°C. The differential pressure was seen to simultaneously return to near zero at the end of the regeneration. Similar observations were repeated for the next four regeneration cycles. It is essential to note that, after each regeneration cycle, the differential pressure always returns to near zero indicating high regeneration efficiencies. Similar regeneration trends were observed for Test 6-165 as shown in Figures 14 and 15. In these two figures, the CO concentration is also plotted, where the peaks of CO are observed to be synchronized with the temperature peaks. The observations are expected since both are associated with soot combustion.

It should be pointed out that the duration of microwave heating between 10 and 5 minutes does not generate any difference on the regeneration of DPF as indicated in Figures 13 and 15. Although not shown, this observation has been confirmed throughout the tests conducted during the Task 4 investigation. The main reason for this observation is that the microwave energy only serves to raise the exhaust gas temperature to reach the ignition temperature of the trapped soot to create soot combustion. The heat generated from soot combustion further raises the exhaust gas temperature to reach its maximum. The temperature then cools down as soon as the soot combustion is completed. It is worth mentioning that, once the soot combustion starts, the continuous supply of heat from microwave power becomes unnecessary since it is insignificant as compared to that generated from soot combustion. The use of 5 minutes of microwave heating, therefore, is sufficient and more energy efficient for microwave regeneration of the developed protocol.

It should also be reported that the original DPF filter was taken out from the holder for examination after 20 microwave regeneration tests including 15 off-line and 5 on-line. No
apparent physical damages on filter cells were observed. The only noticeable difference was that a slight increase in pressure drop was observed as compared to a new filter. An accumulation of soot ash in the DPF wall was suspected to cause this slightly higher pressure drop. Although not shown, the off-line regeneration has been observed to be more efficient in DPF regeneration than on-line regeneration due to its higher oxygen content.

B. Optimal Parameters for NOx Control

As reported in the Task 2 Report, numerous series of NOx/VOC destruction tests have been carried out in the microwave test unit using the constructed prototype control unit shown in Figure 1 with both virgin and regenerated catalysts. It has been generally observed that the NOx (NO + NO2) in the diesel exhaust is successfully reduced with the injection of hexane as a reducing agent. Both the virgin and the regenerated catalysts have shown similar efficiencies. Additional tests have been conducted and the following sections summarize optimal operating conditions based on the results observed.

Optimal DENOX Catalyst Temperature

Many series of NOx reduction tests have been completed in the microwave test facility first using a NO standard of 1045 ppm of NO to characterize the effects of residence time, catalyst bed temperature and hexane injection rate on NO reduction. After many modifications, the current system has been able to achieve an excellent and consistent NO reduction result. A typical set of such results representing the current system are shown in Table 1, where the experiments were conducted under 900°C of bed temperature with three NO flow rates. The hexane injection rate was the same at 10 cm3/min for all the three NO flow rates. The results shown in the table indicate that the NO conversion efficiencies are extremely high at more than 99%. However, the CO oxidation is not as good with higher than 500 ppm still in the treated gas. This CO problem has later been solved as will be reported in the section below. It should be noted that, after numerous tests, it has been concluded that the optimal DENOX bed temperature for the developed catalyst is between 900°C to 1100°C.

In addition to employing NO standards in the NO reduction tests, real engine exhaust has also been involved in the tests. A typical set of such results are shown in Table 2, where the catalyst bed temperature is at 1000°C with four hexane injection rates at 0, 0.1, 0.15, and 0.2 of the engine exhaust. The results again indicate that the NOx reduction efficiencies are excellent at higher than 93% when hexane is injected. Without hexane injection, the efficiency is seen to drop down to about 78%. Among the three hexane injection rates, the one with 10% of engine exhaust appears to generate the best result with a 97.6% of NO reduction efficiency. Again, the results indicate that CO oxidation is not effective with the current system. It is worth reporting that the high temperature in the DENOX catalyst bed may cause a trace amount of SiC to react with oxygen to form CO. The generated CO, however, enhances NO reduction on the catalyst bed as can be seen in a typical set of results shown in Figure 16.
Optimal CO/VOC Catalytic Temperature

As reported in the previous section, the developed catalyst beds have been effective for NO reduction but not for CO oxidation. After many tests and modifications based on literature reports, it has finally been identified that the CO/VOC catalyst was not effective because it was not operated at the optimal temperature. In the previous design, the two catalyst beds, i.e., the DENOX and the CO/VOC beds, were line up in the developed protocol under the same microwave power, which created a higher than needed temperature for the CO/VOC catalyst bed. The CO/VOC bed has since been separated from the DENOX bed in the redesign of microwave waveguide. The optimal temperature for the CO/VOC bed has then been tested to be around 200-400°C and the CO oxidation efficiency is seen to improve dramatically as can be seen from the results shown in Table 3. These results indicate that CO may be generated from the DENOX bed at 436 ppm and the generated CO has subsequently been oxidized in the CO/VOC bed and been decreased to 10 ppm. In addition, the CO/VOC catalyst is capable of reducing the NOx further from 296 ppm down to 3 ppm with the existence of CO. Similar results can be observed in the results shown in Figure 17. These results in the figure indicate that the microwave heating induces the formation of CO from SiC, the formed CO is subsequently decreased to near zero when the CO/VOC catalyst is made on-line, the CO reappears after the CO/VOC is made off-line, the CO level increases further once the hexane is injected, the CO again decreases to zero when the CO/VOC catalyst is made on-line again. While the CO concentration varies, the NO concentration remains low during the entire period when the microwave heating is on. Several additional plots showing typical results from DENOX tests are displayed in Figures 18-20. Figure 18 shows results from a typical de-NOx test involving a standard NO stream of 1021 ppm, while Figures 19 and 20 show typical results associated with de-NOx tests involving actual engine exhaust. As can be seen in the figures, the effectiveness of the using of hexane and catalyst beds in the reduction of NO in the exhaust stream is well-indicated. The optimal hexane flow rate has been determined to be at 2.5% of the engine exhaust flow rate.

Summary of Task 5 Activity - The Development of a Commercialization Plan for the Technology

As stated in the proposal, Task 5 of the project is to develop a commercialization plan for the developed technology. The deliverable date for the task is within 18 month of the signed Notice to Proceed Date as issued by TCEQ. This document describes our commercialization plan as requested by TCEQ. The plan includes the following:

1. Consulting Service – The knowledge gained from the project development has enabled us to provide consulting service to assist the industry in better controlling emissions from diesel engines.

2. Patent Application - Every attempt will be made to apply for a patent or patents from the development. If additional investigations are needed for a successful application, efforts will be continued to seek additional research support from TERC-NTRD or other funding.
agencies, including but not limited to, Texas Air Research Center (TARC) and Houston Advanced Research Center (HARC).

3. Commercialization Partnership – After successful patent applications, potential commercialization partners will be identified and communicated throughout the technology development. Commercialization of the developed technology may be materialized through partnership agreements.

4. Small Business – With patents approved, it may be desirable for the project team to establish a small business to commercialize the developed technology. Advice from Lamar University’s Small Business Development Center will be sought regarding business structure and potential capital funding sources.

In our first effort to conduct technology transfer to the diesel emission control community, we have submitted an abstract reporting our current DPF technology, entitled “Active Regeneration of Diesel Particulate Filter Employing Microwave Heating,” to the NASCRE-2 (North American Symposium in Chemical reaction Engineering 2) Meeting for presentation. The Meeting will be held in Houston from February 4-7, 2007. A copy of the submitted abstract is included below.

Abstract for NASCRE-2 Presentation

Wall-flow diesel particulate filters (DPFs) are considered the most effective devises for the control of diesel particulate emissions. A requirement for the reliable operation of the DPFs, however, is the periodic and/or continuous regeneration of the filters. While microwave heating has been considered a potential active regeneration method for the DPFs, past studies on the technology have identified several technical problems leading to filter failure. The problems are mainly associated with the use of inappropriate filter materials for the microwave system and the generation of local hotspots due to uneven microwave heating, resulting in the physical damage to the filters. The objective of this study was to develop and demonstrate the technology employing a microwave-absorbing filter material coupled with an effective waveguide design for the reliable regeneration of DPFs.

In this study, a well-equipped diesel emission control laboratory was established to conduct the experiments. The experimental facilities included a 6-kw diesel generator, an exhaust flow control system, a diesel particulate filter system, a microwave energy supply system, a soot-sampling system, a differential-pressure measurement system, and a temperature measurement system. In the DPF set up, a silicone carbide wall-flow monolith filter (50mm diameter x 150 mm length, cell density = 200 cpsi, pore size = 20 micron) was enclosed in a quartz filter holder. A commercial 1.4-kw microwave oven was modified to accommodate the quartz holder and a waveguide was engineered to evenly supply the microwave energy to the enclosed filter to achieve filter regeneration. In the experiments, the diesel engine exhaust was lined up to flow through the filter with a fixed flow rate. The microwave regeneration was triggered after a specific soot loading was generated based on the differential pressure drop reading.

The results have indicated that the designed system has been able to achieve uniform temperature profiles both in the radial and the vertical DPF positions. The regeneration of DPF by microwave energy has been observed to be highly efficient. The soot filtration efficiency has
remained to be comparably high after more than five cycles of microwave regeneration with no apparent physical damage to the DPF being observed.

**Concluding Remarks**

The proposed microwave technology for the control of NOx and CO in the exhaust gas stream from stationary diesel engines has been developed and ready for commercial applications. Every attempt will be made to apply for a patent or patents from the development. If additional investigations are needed for a successful patent application, efforts will be continued to seek additional research support from TERC-NTRD or other funding agencies, including but not limited to, Texas Air Research Center (TARC) and Houston Advanced Research Center (HARC). After successful patent applications, potential commercialization partners will be identified and communicated throughout the technology development. Commercialization of the developed technology may be materialized through partnership agreements. With patents approved, it may also be desirable for the project team to establish a small business to commercialize the developed technology. Advice from Lamar University’s Small Business Development Center will be sought regarding business structure and potential capital funding sources.
Table 1.

NOx Reduction Test Involving Standard NO Stream – Effect of NO Flow Rate

<table>
<thead>
<tr>
<th>NO Flow (cc/min)</th>
<th>Bed Temp (°C)</th>
<th>Analysis (Testo 350)</th>
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<td></td>
<td></td>
<td>O2</td>
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<tr>
<td>NO Standard</td>
<td></td>
<td>0.3</td>
</tr>
<tr>
<td>DeNOx + VOC/CO catalyst</td>
<td>100</td>
<td>900</td>
</tr>
<tr>
<td>DeNOx + VOC/CO catalyst</td>
<td>200</td>
<td>900</td>
</tr>
<tr>
<td>DeNOx + VOC/CO catalyst</td>
<td>300</td>
<td>900</td>
</tr>
</tbody>
</table>

Note: Hexane vapor injection remained at a constant rate of 10 cc/min.
Table 2.

NOx Reduction Test Involving Engine Exhaust – Effect of Hexane Injection Rate

<table>
<thead>
<tr>
<th>Exhaust Flow</th>
<th>HC (cc/min)</th>
<th>O2</th>
<th>CO</th>
<th>NO</th>
<th>NO2</th>
<th>NOx</th>
<th>SO2</th>
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</thead>
<tbody>
<tr>
<td>Engine Exhaust</td>
<td>9.2</td>
<td>624</td>
<td>427</td>
<td>362</td>
<td>789</td>
<td>263</td>
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<td></td>
<td>100</td>
<td>0</td>
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<td>1.7</td>
<td>-</td>
<td>52</td>
<td>0</td>
<td>52</td>
</tr>
</tbody>
</table>

Analysis

Note: (1) HC is hexane vapor
(2) Temperature of DeNOx catalyst bed is ~1000 °C
(3) VOC/CO catalyst appears inactive compared with the previous test.
Table 3.

NOx Reduction Test Involving Standard NO Stream – Effect of Catalyst Temperature

<table>
<thead>
<tr>
<th>Catalyst*</th>
<th>NO Std Flow (cc/min)</th>
<th>HC (1) (cc/min)</th>
<th>O2 % ppm</th>
<th>CO ppm</th>
<th>NO ppm</th>
<th>NO2 ppm</th>
<th>NOx ppm</th>
</tr>
</thead>
<tbody>
<tr>
<td>NO Standard</td>
<td>0.2</td>
<td>0</td>
<td>1029</td>
<td>92</td>
<td>1121</td>
<td></td>
<td></td>
</tr>
<tr>
<td>DeNOX</td>
<td>200</td>
<td>0</td>
<td>0.3</td>
<td>436</td>
<td>290</td>
<td>6</td>
<td>296</td>
</tr>
<tr>
<td>CO/VOC</td>
<td>200</td>
<td>0</td>
<td>0.3</td>
<td>10</td>
<td>3</td>
<td>0</td>
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Note: (1) CO/VOC catalyst pack temperature = 219°C
(2) DeNOX pack temperature = 950°C
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NO Reduction and CO Oxidation with and without CO/VOC Catalyst.
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Final Project Summary Report

For the TCEQ NTRD Project

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NTRD Project No. 582-5-70807-0007

Prepared for:
Texas Commission on Environmental Quality
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Executive Summary

This final project summary report is prepared and submitted to TCEQ to fulfill the Task 6 deliverables as stated in Section 2.6.4. of this NTRD (New Technology for Research and Development) project. The Task 6 of the project is described below.

Task 6: REPORTING

2.6. Task statement: The PERFORMING PARTY will prepare and submit monthly and/or quarterly detailed progress reports on the status of this project and a comprehensive final report while ensuring compliance with all TCEQ program requirements.

2.6.1. The PERFORMING PARTY will coordinate all project resources to ensure compliance with NTRD program requirements while providing deliverables on-schedule and on-budget.

2.6.2. The PERFORMING PARTY will generate monthly progress reports and a final report summarizing all aspects of the project based on data from the task completion reports.

2.6.3. Schedule: The PERFORMING PARTY shall submit monthly reports to TCEQ by no later than 15 days after the end of each month. The PERFORMING PARTY shall submit the final report to complete this task within 18 months of the signed Notice to Proceed Date as issued by TCEQ.

2.6.4. Deliverables: The PERFORMING PARTY shall submit monthly progress reports with associated billing statements and a final project summary report to the TCEQ upon completion of this task.

Background

Ozone pollution in Southeast Texas, especially in the Houston/Galveston/Beaumont/Port Arthur airshed, represents one of the worst air quality problems in the nation. The major precursors for ozone formation are NOx and VOC emitted from various industrial, mobile, area and biogenic sources. While the region has adopted control strategies for such emissions from many anthropogenic emission sources, one potential source remaining to be controlled is the emissions from thousands of stationary diesel generators in the region.

Diesel exhaust consists of five primary pollutants, namely NOx, PM, VOC, CO and SO2 [1], where NOx and PM emissions from such engines are especially troublesome from the environmental and health concerns. Control of NOx and PM emissions from diesel engines has been a challenging issue. Wall-flow diesel particulate filters (DPFs) are currently considered the most efficient control technology for diesel particulate matter (DPM) while, for diesel NOx control, the technologies include Exhaust Gas Recirculation (EGR), Selective Catalytic Reduction (SCR) by urea, SCR of NRC (NOx Reducing Catalyst) by hydrocarbons, and ADS (NOx Adsorber) in conjunction with low sulphur (less than 15 ppm) diesel fuel and/or biodiesel.
Most of the technologies are still under intense investigation to improve their control efficiency and operation reliability.

One of the technical issues for reliable operation of DPFs is the effective regeneration of the filters by continuously or periodically removing of the trapped diesel particulate matter. Two methods have been developed to achieve the regeneration. One is the passive regeneration which requires the use of oxidation catalysts upstream from the DPF to convert NO in the diesel exhaust to NO\textsubscript{2} and uses the converted NO\textsubscript{2} to oxidize the trapped DPM (soot) to CO/CO\textsubscript{2} to accomplish regeneration [2]. Since the reaction between NO\textsubscript{2} and soot can occur below 250°C, the temperature of the exhaust gas itself is sufficient to accomplish the regeneration without additional energy. This method is termed “passive” because it uses the energy already in the exhaust gas. The other DPF regeneration method, termed “active” regeneration, involves the supply of additional energy to thermally combust the trapped soot in the presence of oxygen at a temperature higher than 400°C. Since the diesel exhaust system can not consistently generate such a high temperature, additional energy must be used to raise the exhaust gas temperature to accomplish the combustion. This energy may come from additional energy sources such as additional diesel combustion, electric furnaces, electric heating elements, or microwave irradiation [3].

For diesel NO\textsubscript{x} control, as mentioned previously, a promising control technology currently being developed is the use of NRC (NO\textsubscript{x} Reducing Catalyst) to reduce NO\textsubscript{x} to N\textsubscript{2} in the presence of hydrocarbons [4-6]. The technology has been tested on several groups of catalysts, including Cu-ZSM-5, Pt-ZSM-5, Pt-CeO\textsubscript{2}-ZrO\textsubscript{2}, Pt-SiO\textsubscript{2}, Pt-TiO\textsubscript{2}, Pt-Nb\textsubscript{2}O\textsubscript{5}, Pt-WO\textsubscript{3}, Pt-MgO, as well as silver/alumina [7-9]. The reducing capabilities associated with these catalysts have been reported to be a function of many exhaust conditions. Among them, a critical parameter needed to effectively promote the reducing reaction is the elevated temperature, with a specific temperature window for each catalyst. The optimal temperature windows for the reaction have been observed to be between 250 to 500°C depending on the catalysts and exhaust conditions [see, e.g., 7-9]. It is worth pointing out that, in a combined DPF/NRC (diesel particulate filter/NO\textsubscript{x} Reducing Catalyst) technology for simultaneous DPM and NO\textsubscript{x} control, the application of microwave energy serves to provide the needed energy to thermally combust the trapped soot as well as to raise the temperature for enhanced NO\textsubscript{x} reducing reactions in the catalyst bed.

**Objective**

The objective of the proposed project is to develope microwave-assisted diesel emission control technology for simultaneous NO\textsubscript{x}, VOC and DPM (diesel particulate matter) emissions control from stationary diesel generators. The technology modifies and uses commercially-available low-cost microwave units to thermally combust the trapped DPM for reliable DPF regeneration. The supplied microwave energy also serves to effectively raise the exhaust gas temperature for enhanced NO\textsubscript{x} reduction and VOC destruction in the developed catalyst beds. The goals of this proposed project are to construct prototypes involving SiC (silicon carbide)-DPF, SiC-Pt/Rh de-NO\textsubscript{x} catalyst and Al-CO/VOC catalyst and to characterize the constructed prototypes for optimal control of NO\textsubscript{x}, VOC and DPM. Once successfully demonstrated for stationary diesel generators,
the technology will be further developed for treating exhaust from mobile diesel engines to achieve even greater impacts in emissions reduction.

**Constructed Prototype**

The constructed diesel emission control prototype unit is shown in Figure 1. It is a 50mm diameter x 150mm long silicone carbide unit designed to reduce soot, NOx and VOC from a stationary diesel generator exhaust. In the design, diesel soot (solid PM fraction) is removed by the silicone carbide wall-flow monoliths, which are derived from the flow-through catalyst support where channel ends are alternatively plugged to force the gas flow through porous walls acting as filter. The designed prototype has a total material porosity of 45~50% with the medium pore sizes ranging from 10-20 micro meter. Silicone carbide is selected because it is a good microwave receiver for microwave regeneration of the filter. The DeNOx catalyst pack is made out of platinum/rhodium gauze and silicone carbide foam disc, which is capable of reducing NOx to a low level of <5 ppm. The catalyst pack is heated by microwave up to the reaction temperature and hexane is used as a reducing agent to complete NOx reduction followed by VOC removal from the exhaust stream. A regular honeycomb for the catalytic converter is used for VOC/CO reduction since O2 concentration after the DeNOx is <1%. In the operation, the constructed prototype unit is inserted in a quartz holder with the Interam mat to prevent gas bypassing (see Figure 2). Figures 3 through 6 are also included in this report to further describe the operation of the constructed prototype unit where Figure 3 is a schematic diagram of the diesel emission test facility, Figure 4 is a photo picture of the diesel emission test facility, Figure 5 is a photo picture of a diesel emission test with the constructed prototype enclosed in a microwave oven, and Figure 6 shown the waveguide design for the constructed prototype in a microwave oven.

**Soot Filtration and NOx Reduction Efficiencies**

The constructed diesel emission control prototype unit and the diesel emission test facilities shown in Figures 1 through 6 have been used in carrying out the Task 2 experiments. The experiments are designed to evaluate the soot filtration and the NOx reduction efficiencies. While additional experiments are currently still being carried out, this report summarizes the results observed up to this stage. The experimental procedures and the typical results observed for the two investigations, namely, soot filtration efficiency and NOx reduction efficiency, associated with the constructed prototype emission control unit shown in Figure 1 are reported separately in the following two sections.

**A. Soot Filtration**

After the instrument calibration and a couple of shake-down runs, the constructed prototype unit was installed in the system with soot samples before and after the DPF being collected in the sampler. The collected soot samples were weighed by a high-precision microwave balance to determine the filtration efficiency. It was generally observed that a high soot removal efficiency of better than 90% was obtained based on a 10 minute-sampling time. The exhaust gas was also analyzed for O2, NO, NO2, CO and SO2 by the Testo-350 Gas Analyzer. A typical set of exhaust gas conditions is given in Table 1 for reference.
B. NOx Reduction

Numerous series of NOx reduction tests have been carried out in the microwave test unit using the constructed prototype control unit shown in Figure 1 with both virgin and regenerated catalysts. It has been generally observed that the NOx (NO + NO2) in the diesel exhaust is successfully reduced with the injection of hexane as a reducing agent. It is worth pointing out again that the designed catalyst pack includes an activated platinum gauze (wire mesh), a SiC foam discs (10 mm dia x 4 mm thick, 80 ppi), and followed by a VOC oxidation catalyst (honeycomb). Both the virgin and the regenerated catalysts have shown similar efficiencies. A typical set of experimental data is provided in Table 2 for reference.

Microwave Regeneration and NOx/VOC/Soot Destruction Efficiencies

As described in the submitted Task 3 Report, the constructed diesel emission control prototype unit and the diesel emission test facilities shown in Figures 1 through 6 have been used in carrying out the Task 3 experiments. The experiments are designed to evaluate the efficiencies of microwave regeneration and destruction of NOx/VOC/Soot upon microwave irradiation. The experimental procedures and typical results observed for the two investigations, namely, microwave regeneration efficiency and NOx/VOC/Soot destruction efficiency, associated with the constructed prototype emission control unit shown in Figure 1 are reported separately in the two sections below.

A. Microwave Regeneration

Microwave regeneration of the constructed diesel emission control prototype unit involves the use of microwave energy to oxide the filtered solid particulates (soot) to gaseous products (CO and CO2) at elevated temperatures of 500-600 °C. Since silicone carbide (SiC) exhibits high RF absorption properties (high dielectric constant), it can be effectively heated by microwave. In addition, since the soot has good microwave absorption properties, the heating of soot by the microwave energy to induce combustion is effective as well. In our experiments, the removal of soot has been performed either continuously during regular operation of the prototype unit (on-line regeneration), or periodically after a pre-determined quantity of soot has been accumulated (off-line regeneration). Numerous regeneration tests have been carried out using the constructed prototype unit. During the tests, the temperature of the filter is monitored by a temperature sensor located in the center of the filter. Pressure drop across the filter, CO concentration and filter temperature are simultaneously recorded in the data acquisition system. Typical results are shown in the following sections.

Radial Temperature Profiles

Figure 7 displays typical radial temperature profiles in the constructed prototype unit. As indicated, the radial temperature profiles are relatively uniform within about 100°F during microwave regeneration.
Vertical Temperature Profiles

Figure 8 displays typical vertical temperature profiles in the constructed prototype unit. Again, the vertical temperature profiles are also uniform within about 100°F during microwave regeneration.

Pressure Drop during Regeneration

Figure 9 displays typical pressure drop data during microwave regeneration for a plugged and an unplugged unit. The results indicated the regeneration is successfully completed at the end of the process.

Temperature vs CO Plot

Figure 10 shows a typical plot of temperature vs CO Concentration during Microwave Regeneration. It indicates that the CO concentration jumps up at the temperature of 300°C indicating the start of the soot combustion process.

Temperature vs Pressure Drop Plot

Figure 11 is a typical plot of temperature vs pressure drop during Microwave Regeneration. As indicated, the pressure drop starts to decrease at a temperature of 400°C and eventually returns to the pressure drop corresponding to a clean unit, which indicates complete regeneration.

Optimum Regeneration Duration

The regeneration tests conducted so far have indicated that majority of the soot is burnt off in the first 5 minutes and at > 400 °C, and while maintaining DPF temperature at 600-650°C for remaining 15 minutes, slight changes in the filter DP (differential pressure) and CO concentration have been observed. These tests suggest an optimum regeneration time of 10 minutes with air.

B. NOx/VOC/Soot Destruction

The results shown above have demonstrated the soot destruction capability of the proposed microwave regeneration technology. As reported in the Task 2 Report, numerous series of NOx/VOC destruction tests have been carried out in the microwave test unit using the constructed prototype control unit shown in Figure 1 with both virgin and regenerated catalysts. It has been generally observed that the NOx (NO + NO2) in the diesel exhaust is successfully reduced with the injection of hexane as a reducing agent. Both the virgin and the regenerated catalysts have shown similar efficiencies. The VOC destruction efficiency is expected to be high but has not been confirmed due to the limitation of the Testo 350 Analyzer.
Optimal Design and Operation Parameters

As described in the submitted Task 4 Report, the constructed diesel emission control prototype unit and the diesel emission test facilities shown in Figures 1 through 6 have continuously been used in carrying out the Task 4 experiments. These experiments are designed to evaluate the optimal design and operation parameters associated with the developed technology. The experimental procedures and typical results observed are summarized separately in the following two sections.

A. Optimal Parameters for DPF Regeneration

As described previously, microwave regeneration of the constructed diesel emission control prototype unit involves the use of microwave energy to oxide the filtered solid particulates (soot) to gaseous products (CO and CO₂) at elevated temperatures of 500-600 °C. Since silicone carbide (SiC) exhibits high RF absorption properties (high dielectric constant), it can be effectively heated by microwave. In addition, since the soot has good microwave absorption properties, the heating of soot by the microwave energy to induce combustion is effective as well. In our experiments, the removal of soot has been performed either continuously during regular operation of the prototype unit (on-line regeneration), or periodically after a pre-determined quantity of soot has been accumulated (off-line regeneration). Numerous regeneration tests have been carried out using the constructed prototype unit. During the tests, the temperature of the filter is monitored by a temperature sensor located in the center of the filter. Pressure drop across the filter, CO concentration and filter temperature are simultaneously recorded in the data acquisition system. Summarized results are reported below.

Soot Loading Rate

To effectively investigate optimal parameters for DPFR regeneration, a new control valve was installed in the DPF regeneration facility for maintaining a constant exhaust gas flow rate during the regeneration tests. The modification enabled the project to accurately estimate the soot loading rate through measuring the differential pressure drop of DPF at a particulate flow rate. A series of tests were performed and a typical set of correlation results between the soot loading rate and the differential pressure drop reading is given in Figure 12. As indicated in Figure 12, the soot loading rate is seen to be proportional to the measured differential pressure drop across the DPF. The observed correlation allows the project to estimate the amount of soot loading on the DPF based on the measured differential pressure drop.

Microwave DPF Regeneration

Numerous sets of DPF regeneration tests were conducted to investigate the DPF regeneration characteristics involving microwave heating. Experimental observations from three typical regeneration tests (5 cycles for each test) are displayed in Figures 13-15, where Figure 13 is for Test 6-163 with a 10 minutes regeneration time and Figures 14 and 15 are for Test 6-165 with a 5 minutes regeneration time. The results shown in Figure 13 indicate that, once the engine was turned on, the differential pressure started to increase due to soot loading and the increase in temperature. This pressure reached to about 50 inch of H₂O in about 20 minutes. During the
period, the exhaust gas temperature was also seen to increase from room temperature to about 250 °C. No soot combustion was observed at this temperature because it was below the soot ignition temperature. At this moment when the differential pressure reached 50 inch of H2O, the microwave power was turned on and the exhaust gas temperature was seen to rise above the soot ignition temperature at about 400°C. This elevated temperature ignited the soot and the heat generated from soot combustion further elevates the gas temperature to its maximum of higher than 700°C. The differential pressure was seen to simultaneously return to near zero at the end of the regeneration. Similar observations were repeated for the next four regeneration cycles. It is essential to note that, after each regeneration cycle, the differential pressure always returns to near zero indicating high regeneration efficiencies. Similar regeneration trends were observed for Test 6-165 as shown in Figures 14 and 15. In these two figures, the CO concentration is also plotted, where the peaks of CO are observed to be synchronized with the temperature peaks. The observations are expected since both are associated with soot combustion.

It should be pointed out that the duration of microwave heating between 10 and 5 minutes does not generate any difference on the regeneration of DPF as indicated in Figures 13 and 15. Although not shown, this observation has been confirmed throughout the tests conducted during the Task 4 investigation. The main reason for this observation is that the microwave energy only serves to raise the exhaust gas temperature to reach the ignition temperature of the trapped soot to create soot combustion. The heat generated from soot combustion further raises the exhaust gas temperature to reach its maximum. The temperature then cools down as soon as the soot combustion is completed. It is worth mentioning that, once the soot combustion starts, the continuous supply of heat from microwave power becomes unnecessary since it is insignificant as compared to that generated from soot combustion. The use of 5 minutes of microwave heating, therefore, is sufficient and more energy efficient for microwave regeneration of the developed protocol.

It should also be reported that the original DPF filter was taken out from the holder for examination after 20 microwave regeneration tests including 15 off-line and 5 on-line. No apparent physical damages on filter cells were observed. The only noticeable difference was that a slight increase in pressure drop was observed as compared to a new filter. An accumulation of soot ash in the DPF wall was suspected to cause this slightly higher pressure drop. Although not shown, the off-line regeneration has been observed to be more efficient in DPF regeneration than on-line regeneration due to its higher oxygen content.

B. Optimal Parameters for NOx Control

As reported in the Task 2 Report, numerous series of NOx/VOC destruction tests have been carried out in the microwave test unit using the constructed prototype control unit shown in Figure 1 with both virgin and regenerated catalysts. It has been generally observed that the NOx (NO + NO2) in the diesel exhaust is successfully reduced with the injection of hexane as a reducing agent. Both the virgin and the regenerated catalysts have shown similar efficiencies. Additional tests have been conducted and the following sections summarize optimal operating conditions based on the results observed.
Optimal DENOX Catalyst Temperature

Many series of NOx reduction tests have been completed in the microwave test facility first using a NO standard of 1045 ppm of NO to characterize the effects of residence time, catalyst bed temperature and hexane injection rate on NO reduction. After many modifications, the current system has been able to achieve an excellent and consistent NO reduction result. A typical set of such results representing the current system are shown in Table 1, where the experiments were conducted under 900°C of bed temperature with three NO flow rates. The hexane injection rate was the same at 10 cm³/min for all the three NO flow rates. The results shown in the table indicate that the NO conversion efficiencies are extremely high at more than 99%. However, the CO oxidation is not as good with higher than 500 ppm still in the treated gas. This CO problem has later been solved as will be reported in the section below. It should be noted that, after numerous tests, it has been concluded that the optimal DENOX bed temperature for the developed catalyst is between 900°C to 1100°C.

In addition to employing NO standards in the NO reduction tests, real engine exhaust has also been involved in the tests. A typical set of such results are shown in Table 2, where the catalyst bed temperature is at 1000°C with four hexane injection rates at 0, 0.1, 0.15, and 0.2 of the engine exhaust. The results again indicate that the NOx reduction efficiencies are excellent at higher than 93% when hexane is injected. Without hexane injection, the efficiency is seen to drop down to about 78%. Among the three hexane injection rates, the one with 10% of engine exhaust appears to generate the best result with a 97.6% of NO reduction efficiency. Again, the results indicate that CO oxidation is not effective with the current system. It is worth reporting that the high temperature in the DENOX catalyst bed may cause a trace amount of SiC to react with oxygen to form CO. The generated CO, however, enhances NO reduction on the catalyst bed as can be seen in a typical set of results shown in Figure 16.

Optimal CO/VOC Catalytic Temperature

As reported in the previous section, the developed catalyst beds have been effective for NO reduction but not for CO oxidation. After many tests and modifications based on literature reports, it has finally been identified that the CO/VOC catalyst was not effective because it was not operated at the optimal temperature. In the previous design, the two catalyst beds, i.e., the DENOX and the CO/VOC beds, were line up in the developed protocol under the same microwave power, which created a higher than needed temperature for the CO/VOC catalyst bed. The CO/VOC bed has since been separated from the DENOX bed in the redesign of microwave waveguide. The optimal temperature for the CO/VOC bed has then been tested to be around 200-400°C and the CO oxidation efficiency is seen to improve dramatically as can be seen from the results shown in Table 3. These results indicate that CO may be generated from the DENOX bed at 436 ppm and the generated CO has subsequently been oxidized in the CO/VOC bed and been decreased to 10 ppm. In addition, the CO/VOC catalyst is capable of reducing the NOx further from 296 ppm down to 3 ppm with the existence of CO. Similar results can be observed in the results shown in Figure 17. These results in the figure indicate that the microwave heating induces the formation of CO from SiC, the formed CO is subsequently decreased to near zero when the CO/VOC catalyst is made on-line, the CO reappears after the CO/VOC is made off-line, the CO level increases further once the hexane is injected, the CO
again decreases to zero when the CO/VOC catalyst is made on-line again. While the CO concentration varies, the NO concentration remains low during the entire period when the microwave heating is on. Several additional plots showing typical results from DENOX tests are displayed in Figures 18-20. Figure 18 shows results from a typical de-NOx test involving a standard NO stream of 1021 ppm, while Figures 19 and 20 show typical results associated with de-NOx tests involving actual engine exhaust. As can be seen in the figures, the effectiveness of the using of hexane and catalyst beds in the reduction of NO in the exhaust stream is well-indicated. The optimal hexane flow rate has been determined to be at 2.5% of the engine exhaust flow rate.

**Development of a Commercialization Plan for the Technology**

As stated in the proposal, Task 5 of the project is to develop a commercialization plan for the developed technology. The deliverable date for the task is within 18 month of the signed Notice to Proceed Date as issued by TCEQ. This document describes our commercialization plan as requested by TCEQ. The plan includes the following:

1. **Consulting Service** – The knowledge gained from the project development has enabled us to provide consulting service to assist the industry in better controlling emissions from diesel engines.

2. **Patent Application** - Every attempt will be made to apply for a patent or patents from the development. If additional investigations are needed for a successful application, efforts will be continued to seek additional research support from TERC-NTRD or other funding agencies, including but not limited to, Texas Air Research Center (TARC) and Houston Advanced Research Center (HARC).

3. **Commercialization Partnership** – After successful patent applications, potential commercialization partners will be identified and communicated throughout the technology development. Commercialization of the developed technology may be materialized through partnership agreements.

4. **Small Business** – With patents approved, it may be desirable for the project team to establish a small business to commercialize the developed technology. Advice from Lamar University’s Small Business Development Center will be sought regarding business structure and potential capital funding sources.

In our first effort to conduct technology transfer to the diesel emission control community, we have submitted an abstract reporting our current DPF technology, entitled “Active Regeneration of Diesel Particulate Filter Employing Microwave Heating,” to the NASCRE-2 (North American Symposium in Chemical reaction Engineering 2) Meeting for presentation. The Meeting will be held in Houston from February 4-7, 2007. A copy of the submitted abstract is included below.
Abstract for NASCRE-2 Presentation

Wall-flow diesel particulate filters (DPFs) are considered the most effective devices for the control of diesel particulate emissions. A requirement for the reliable operation of the DPFs, however, is the periodic and/or continuous regeneration of the filters. While microwave heating has been considered a potential active regeneration method for the DPFs, past studies on the technology have identified several technical problems leading to filter failure. The problems are mainly associated with the use of inappropriate filter materials for the microwave system and the generation of local hotspots due to uneven microwave heating, resulting in the physical damage to the filters. The objective of this study was to develop and demonstrate the technology employing a microwave-absorbing filter material coupled with an effective waveguide design for the reliable regeneration of DPFs.

In this study, a well-equipped diesel emission control laboratory was established to conduct the experiments. The experimental facilities included a 6-kw diesel generator, an exhaust flow control system, a diesel particulate filter system, a microwave energy supply system, a soot-sampling system, a differential-pressure measurement system, and a temperature measurement system. In the DPF set up, a silicone carbide wall-flow monolith filter (50mm diameter x 150 mm length, cell density = 200 cpsi, pore size = 20 micron) was enclosed in a quartz filter holder. A commercial 1.4-kw microwave oven was modified to accommodate the quartz holder and a waveguide was engineered to evenly supply the microwave energy to the enclosed filter to achieve filter regeneration. In the experiments, the diesel engine exhaust was lined up to flow through the filter with a fixed flow rate. The microwave regeneration was triggered after a specific soot loading was generated based on the differential pressure drop reading.

The results have indicated that the designed system has been able to achieve uniform temperature profiles both in the radial and the vertical DPF positions. The regeneration of DPF by microwave energy has been observed to be highly efficient. The soot filtration efficiency has remained to be comparably high after more than five cycles of microwave regeneration with no apparent physical damage to the DPF being observed.

Summary of Task 6 Activity - Reporting

The project PI has prepared and submitted monthly progress reports on the status of the project and this comprehensive final report while ensuring compliance with all TCEQ program requirements.

1. The project PI has coordinated all project resources to ensure compliance with NTRD program requirements while providing deliverables on-schedule and on-budget.

2. The project PI has generated monthly progress reports and a final report summarizing all aspects of the project based on data from the task completion reports.

3. Schedule: The Project PI has submitted monthly reports to TCEQ by no later than 15 days after the end of each month. The project PI has submitted this final report to
complete this task within 18 months of the signed Notice to Proceed Date as issued by TCEQ.

4. Deliverables: The project PI has submitted monthly progress reports with associated billing statements and a final project summary report to the TCEQ upon completion of this task.

Conclusions

The project PI has coordinated all project resources to ensure compliance with NTRD program requirements while providing deliverables on-schedule and on-budget. He has generated monthly progress reports and a final report summarizing all aspects of the project based on data from the task completion reports. In conclusion, the proposed microwave technology for the control of NOx and CO in the exhaust gas stream from stationary diesel engines has been developed and ready for commercial applications. The developed DPF regeneration technology employing microwave heating is effective and energy efficient. The required microwave heating is only to raise the exhaust gas temperature to the ignition temperature of the trapped soot at about 400°C. The heat generated from soot combustion will then complete the regeneration process. In the developed protocol, a 5 minute of microwave heating is sufficient to induce soot combustion for each regeneration cycle. The modified catalyst beds design in the protocol is seen to produce an effective and consistent control of both NOx and CO from diesel exhaust. The optimal bed temperatures for the two catalyst beds appear to be 900-1100°C for the DENOX bed and 200-400°C for the CO/VOC bed. The injection of hexane enhances the NO reduction efficiency while, even without hexane injection, the high temperature bed may promote the formation of CO from SiC, which results in the enhancing of NO reduction on the DENOX and CO/VOC catalysts. The optimal hexane flow rate has been determined to be at 2.5% of the engine exhaust flow rate. Every attempt will be made to apply for a patent or patents from the development. If additional investigations are needed for a successful patent application, efforts will be continued to seek additional research support from TERC-NTRD or other funding agencies, including but not limited to, Texas Air Research Center (TARC) and Houston Advanced Research Center (HARC). After successful patent applications, potential commercialization partners will be identified and communicated throughout the technology development. Commercialization of the developed technology may be materialized through partnership agreements. With patents approved, it may also be desirable for the project team to establish a small business to commercialize the developed technology. Advice from Lamar University’s Small Business Development Center will be sought regarding business structure and potential capital funding sources.
Literature Cited


### Table 1. Soot Sampling Conditions

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<th>Exhaust (m³/hr)</th>
<th>Load (amp)</th>
<th>Temperature Inlet°C</th>
<th>Temperature Outlet°C</th>
<th>Press &quot;H₂O (ppm)</th>
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Note: 1. Soot samples were taken after 1 hour warm up at 70% of electric load.
2. CO₂ concentration in %, and others in ppm.
Table 2.

Typical NOx Reduction Test

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<td>Watt</td>
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<td>(%)</td>
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<td>Exhaust with DeNOx/VOC Catalyst</td>
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Note: Analysis done by Testo 350 Analyzer
Table 3.

NOx Reduction Test Involving Standard NO Stream – Effect of NO Flow Rate

<table>
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<tr>
<th>NO Flow (cc/min)</th>
<th>Bed Temp (°C)</th>
<th>O2</th>
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</tr>
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<td>DeNOx + VOC/CO catalyst</td>
<td>100</td>
<td>900</td>
<td>0.2</td>
<td>4800</td>
<td>11</td>
<td>0</td>
</tr>
<tr>
<td>DeNOx + VOC/CO catalyst</td>
<td>200</td>
<td>900</td>
<td>0.2</td>
<td>1114</td>
<td>20</td>
<td>1</td>
</tr>
<tr>
<td>DeNOx + VOC/CO catalyst</td>
<td>300</td>
<td>900</td>
<td>0.2</td>
<td>366</td>
<td>26</td>
<td>0</td>
</tr>
</tbody>
</table>

Note: Hexane vapor injection remained at a constant rate of 10 cc/min.
Table 4.

NOx Reduction Test Involving Engine Exhaust – Effect of Hexane Injection Rate

<table>
<thead>
<tr>
<th>Exhaust Flow (cc/min)</th>
<th>HC (cc/min)</th>
<th>O2 ppm</th>
<th>CO ppm</th>
<th>NO ppm</th>
<th>NO2 ppm</th>
<th>NOx ppm</th>
<th>SO2 ppm</th>
</tr>
</thead>
<tbody>
<tr>
<td>Engine Exhaust</td>
<td></td>
<td>9.2</td>
<td>624</td>
<td>427</td>
<td>362</td>
<td>789</td>
<td>263</td>
</tr>
<tr>
<td>100</td>
<td>0</td>
<td>2.5</td>
<td>8375</td>
<td>144</td>
<td>28</td>
<td>172</td>
<td>-</td>
</tr>
<tr>
<td>100</td>
<td>10</td>
<td>0.9</td>
<td>28322</td>
<td>19</td>
<td>0</td>
<td>19</td>
<td>-</td>
</tr>
<tr>
<td>100</td>
<td>15</td>
<td>1.1</td>
<td>40309</td>
<td>22</td>
<td>0</td>
<td>22</td>
<td>-</td>
</tr>
<tr>
<td>100</td>
<td>20</td>
<td>1.7</td>
<td>-</td>
<td>52</td>
<td>0</td>
<td>52</td>
<td>-</td>
</tr>
</tbody>
</table>

Note: (1). HC is hexane vapor
(2) Temperature of DeNOx catalyst bed is ~1000 °C
(3) VOC/CO catalyst appears inactive compared with the previous test.
Table 5. NOx Reduction Test Involving Standard NO Stream – Effect of Catalyst Temperature

<table>
<thead>
<tr>
<th>Catalyst*</th>
<th>NO Std Flow (cc/min)</th>
<th>HC(^{(1)}) (cc/min)</th>
<th>O2</th>
<th>CO</th>
<th>NO</th>
<th>NO2</th>
<th>NOx</th>
</tr>
</thead>
<tbody>
<tr>
<td>NO Standard</td>
<td>0.2</td>
<td>0</td>
<td>1029</td>
<td>92</td>
<td>1121</td>
<td></td>
<td></td>
</tr>
<tr>
<td>DeNO(\text{X})</td>
<td>200</td>
<td>0</td>
<td>0.3</td>
<td>436</td>
<td>290</td>
<td>6</td>
<td>296</td>
</tr>
<tr>
<td>CO/VOC</td>
<td>200</td>
<td>0</td>
<td>0.3</td>
<td>10</td>
<td>3</td>
<td>0</td>
<td>3</td>
</tr>
</tbody>
</table>

Note:  
(1) CO/VOC catalyst pack temperature = 219°C  
(2) DeNO\(\text{X}\) pack temperature = 950°C  
(3) NO standard is 1023 ppm NO in nitrogen
Figure 1.

The Current Constructed Diesel Emission Control Prototype Unit.
Figure 2.
Schematic Diagram of the Designed Holder for the Constructed Prototype.
Figure 3.

Figure 4.

Photo Picture of the Diesel Emission Test Facility.
Figure 5.

Diesel Emission Test with the Constructed Prototype Enclosed in a Microwave Oven.
Figure 6.

The Waveguide Design for the Constructed Prototype.
Figure 7.

Typical Radial Temperature Profile in the Prototype Unit.
Figure 8.

Typical Vertical Temperature Profile in the Prototype Unit.
Figure 9.

Pressure Drop Profiles Associated with Plugged and Clean Prototype Units.
Figure 10.

Plot of Temperature vs CO Concentration during Microwave Regeneration.
Figure 11.

Plot of Temperature vs Pressure Drop during Microwave Regeneration.
Figure 12.

Plot of DPF Pressure Drop during Engine Operation.
Figure 13.

Typical 5-cycle Soot Loading/Regeneration Tests
(Test 6-163; Microwave Heating Duration: 10 minutes).
Exhaust Flow = 5m³/hr
Regen Air = 10 l/min
Regen Time = 5 min
MW = 1400 kw

Figure 14.
Typical 5-cycle Soot Loading/Regeneration Tests
(Test 6-165; Microwave Heating Duration: 5 minutes).
Figure 15.

Typical 5-cycle Soot Loading/Regeneration Tests Corresponding to Figure 14 (Test 6-165; Microwave Heating Duration: 5 minutes).
Figure 16.

NOx Reduction and Formation of CO from SiC due to Microwave Heating.
Figure 17.

NO Reduction and CO Oxidation with and without CO/VOC Catalyst.
Figure 18.

Plot of Temperature and NO/CO Concentrations during a De-NOx Test Involving a Standard NO Stream of 1021 ppm.
(Note: MW-Microwave; DOC-Diesel Oxidation Catalyst)
Figure 19.

Effect of HC Flow Rate on NO and CO Concentrations during a De-NOx Test Involving Actual Engine Exhaust.
(Note: MW-Microwave; HC-Hydrocarbon; DOC-Diesel Oxidation Catalyst)
Figure 20.

Effects of DOC and HC (Hexane) on NO and CO Concentrations during a De-NOx Test Involving Actual Engine Exhaust.
(Note: MW-Microwave; HC-Hydrocarbon; DOC-Diesel Oxidation Catalyst)