Emission performance of lignin-derived cyclic oxygenates in a heavy-duty diesel engine
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ABSTRACT

In earlier research, a new class of bio-fuels, so-called cyclic oxygenates, was reported to have a favorable impact on the soot-\(\text{NO}_x\) trade-off experience in diesel engines. In this paper, the soot-\(\text{NO}_x\) trade-off is compared for two types of cyclic oxygenates. 2-phenyl ethanol has an aromatic and cyclohexane ethanol a saturated or aliphatic ring structure. Accordingly, the research is focused on the effect of aromaticity on the aforementioned emissions trade-off. This research is relevant because, starting from lignin, a biomass component with a complex poly-aromatic structure, the production of 2-phenyl ethanol requires less hydrogen and can therefore be produced at lower cost than is the case for cyclohexane ethanol. The goal of this paper, realized by means of experiments on a modified DAF heavy-duty diesel engine, is to investigate whether or not the (potentially prohibitively) expensive hydrogenation step from 2-phenyl ethanol to cyclohexane ethanol has an added value from an emissions perspective. The results suggest that this is not the case and hydrogenation therefore does not seem like an interesting additional step in the production process.

INTRODUCTION

Diesel engines are known to offer good fuel economy and low carbon dioxide (\(\text{CO}_2\)) emissions. Unfortunately, the diesel engine is a source of soot and \(\text{NO}_x\) emissions, both of which are subject to legal limits because of their adverse effects on the environment and human health. Moreover, soot emissions from diesel engines produce a significant fraction of urban airborne particulate matter, which has been suspected to cause an increased incidence of respiratory problems and cancer. It is therefore of considerable interest to reduce soot escaping oxidation during the diesel combustion process. In order to meet the requirements of future legislation, emissions of these substances, as well as those of carbon monoxide (\(\text{CO}\)) and unburnt hydrocarbons (\(\text{HC}\)), will have to be reduced. This could be accomplished either by improving the combustion process within the cylinder or by other means, for example by using alternative fuels with oxygenated compounds.

In order to realize lower \(\text{NO}_x\) and soot emissions, improvements of fuel injection systems (e.g. rate shaping, multiple injections), optimized combustion chamber designs and exhaust gas recirculation (EGR) have been investigated \[1,2,3\]. Still, Diesel engines produce relatively high \(\text{NO}_x\) and soot emissions that require expensive and, often, fuel consuming exhaust gas after treatment technology. This is the driver for continuous research, heading towards alternative combustion modes, which promise a drastic reduction of engine-out emissions at still favorable fuel efficiency.

In addition, there have been many studies on fuel effects on exhaust emissions, especially with respect to soot \[4,5\]. The results suggest that reductions in sulfur, density, distillation temperature and aromatic content, in particular multi-ring variants, lower soot emissions. Although fuel properties are the main factors influencing engine performance and emissions, there are still many challenges to identify the optimal fuel properties and their effects on engine behavior. In particular, many researchers have investigated the relationship between chemical properties of fuels and formation of pollutants. The pyrolysis and oxidation of several light hydrocarbon fuels was investigated over a wide temperature range using shock tubes \[6,7\]. The thermal decomposition of several aliphatic hydrocarbon fuels was also investigated using a fluid reaction tube \[8\]. These authors reported the formation mechanism of benzene rings from the decomposed low-boiling point hydrocarbons. Among various fuel properties such as CN, distillation
characteristics, aromatic content, the aromatic content in particular is known to have significant effect on NO\textsubscript{x} and soot emissions \[14\].

Because of the depletion of conventional oil, future transportation fuels are expected to contain an increasing portion of alternative fuel components. Consequently, a deeper understanding of the combustion process not only of normal diesel, but also of possible alternative fuels, could be of help in reaching stringent emission limits with maintained efficiency. A number of studies have shown substantial soot reductions for biodiesel and biodiesel blended with petroleum diesel \[9, 10\], relative to neat petroleum diesel. However, they also show a slight increase in NO\textsubscript{x} emissions. Further research has shown that the molecular structure of biodiesel could have a substantial impact on emissions. Recent studies on fuel properties, especially CN, indicate that the homogeneity of the cylinder charge is related to the CN of diesel or diesel-like fuels \[11\]. Low CN fuels that are resistant to ignite create longer ignition delays, thereby resulting in extended mixing time for fuel and air \[12, 13\]. Yet, the strong resistance to auto-ignition of the low CN fuels may cause difficulties in certain combustion conditions, such as cold start \[14\].

A strong connection between fuel molecular structure and the soot formation mechanism during diesel combustion has been shown and discussed in some fundamental studies \[15,16,17\]. Basic studies on soot formation in laminar diffusion flames have revealed that the sooting tendency of most hydrocarbons is profoundly affected by the different types of carbon-carbon bonds \[15\]. In general, compared with straight chain hydrocarbons, soot production of fuels with aromatic rings and triple-bonded carbon-carbon are relatively high \[15,16,17\]. While some studies \[18,19,20\] have focused on the effect of aromatics on engine exhaust emissions, variations of aromatic content are often accompanied by changes in other fuel properties, such as oxygen fraction, CN, boiling behavior, viscosity and heating value. It is therefore quite difficult to isolate the effect of aromaticity as such with respect to engine performance and pollutant emissions.

Starting from lignin, which has a complex poly-aromatic structure, the production of aromatic compounds (e.g. 2-phenyl ethanol) requires less hydrogen and can therefore be produced at lower cost than is the case for saturated or aliphatic compounds (e.g. cyclohexane ethanol). The goal of this paper is to investigate whether or not the hydrogenation step from 2-phenyl ethanol to cyclohexane ethanol has an added value with respect to fuel economy and/or the soot-NO\textsubscript{x} trade-off.

**MATERIALS AND METHODS**

**FUEL PREPARATION**

The physical properties and heating values of the neat base fuel and oxygenates are listed in the table below.

As shown in Table 2, the CN of the cyclic oxygenates has been derived from the measured MON (ASTM D2700 \[22\]) and RON (ASTM2699 \[23\]) values of blends with gasoline. First, the MON and RON values of the pure oxygenates are calculated according to Kay's mixing rule \[24\]. Finally, the corresponding CN of these two fuels follows from equation (1) proposed by Kalghatgi \[25\].

\[
CN = 54.6 - 0.42 \text{ RON}
\] (1)

Both oxygenates are blended to commercial diesel (EN590) to equal a fuel oxygen content of 2.33 (Table 3), as it is well-known that fuel oxygen heavily influences the soot-NO\textsubscript{x} trade-off \[14\].

**MEASUREMENT SETUP**

The test engine, Cyclops \[26\], is a dedicated test rig, designed and built at the Eindhoven University of Technology. It is based on a DAF XE 355 C engine. The specifications of this engine are presented in Table 4. Cylinders 4 thru 6 operate under the stock engine control unit, and together with a water-cooled, eddy-current Schenck W450 dynamometer, they are only used to start the engine and control the rotational speed of the test cylinder, i.e. cylinder 1.
### Table 2. CN derivation for the cyclic oxygenates

<table>
<thead>
<tr>
<th>Name of Fuel/Blend</th>
<th>Diesel (EN590)</th>
<th>Gasoline 90% &amp; 10% 2-Phenyl ethanol</th>
<th>90% Gasoline 10% Cyclohexane ethanol</th>
<th>2-Phenyl ethanol</th>
<th>Cyclohexane ethanol</th>
</tr>
</thead>
<tbody>
<tr>
<td>Molecular formula</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>C₆H₁₀O</td>
</tr>
<tr>
<td>Measured MON [22]</td>
<td>-</td>
<td>85.6</td>
<td>86.1</td>
<td>81.8</td>
<td>-</td>
</tr>
<tr>
<td>Measured RON [23]</td>
<td>-</td>
<td>95.0</td>
<td>96.9</td>
<td>90.1</td>
<td>-</td>
</tr>
<tr>
<td>Calculated MON [24]</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>89.5</td>
</tr>
<tr>
<td>Calculated RON [24]</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>110</td>
<td>51.3</td>
</tr>
<tr>
<td>Evaluated CN</td>
<td>56 (according to IP 498)</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>8.5</td>
</tr>
</tbody>
</table>

### Table 3. Test blend properties

<table>
<thead>
<tr>
<th>Name of Blend</th>
<th>Phenyl ethanol-15%</th>
<th>Cyclohexane ethanol-17.1%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Base Fuel</td>
<td>Diesel</td>
<td>Diesel</td>
</tr>
<tr>
<td>Base Fuel [vol.-%]</td>
<td>85</td>
<td>82.9</td>
</tr>
<tr>
<td>Oxygenate</td>
<td>Phenyl ethanol</td>
<td>Cyclohexane ethanol</td>
</tr>
<tr>
<td>Concentration [vol.-%]</td>
<td>15</td>
<td>17.1</td>
</tr>
<tr>
<td>Oxygen [wt.-%]</td>
<td>2.33</td>
<td>2.33</td>
</tr>
<tr>
<td>Density [kg/l]</td>
<td>0.858</td>
<td>0.843</td>
</tr>
<tr>
<td>Calculated LHV [MJ/kg]</td>
<td>40.4</td>
<td>40.7</td>
</tr>
<tr>
<td>LHV [MJ/kg] vs. diesel [%]</td>
<td>-2.7</td>
<td>-1.9</td>
</tr>
<tr>
<td>Calculated LHV [MJ/l]</td>
<td>34.7</td>
<td>34.3</td>
</tr>
<tr>
<td>LHV [MJ/l] vs. diesel [%]</td>
<td>+0.7</td>
<td>-0.4</td>
</tr>
<tr>
<td>Calculated CN [-]</td>
<td>42.3</td>
<td>49.3</td>
</tr>
<tr>
<td>CN vs. diesel [-]</td>
<td>-13.7</td>
<td>-6.7</td>
</tr>
</tbody>
</table>
Table 4. Cyclops specifications

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Specification</th>
</tr>
</thead>
<tbody>
<tr>
<td>Base engine</td>
<td>6 cylinder HDDI diesel</td>
</tr>
<tr>
<td>Test Cylinder</td>
<td>1 isolated for combustion studies</td>
</tr>
<tr>
<td>Bore [mm]</td>
<td>130</td>
</tr>
<tr>
<td>Stroke [mm]</td>
<td>158</td>
</tr>
<tr>
<td>Compression ratio [-]</td>
<td>15</td>
</tr>
<tr>
<td>Bowl shape</td>
<td>M-shaped</td>
</tr>
<tr>
<td>Bowl diameter [mm]</td>
<td>100</td>
</tr>
</tbody>
</table>

When data acquisition is idle, for instance during engine warm-up or in between measurements, only the three propelling cylinders are fired. Once warmed up and operating at the desired engine speed, combustion phenomena and emission formation can be studied in the test cylinder. Apart from the shared cam- and crankshaft and the lubrication and coolant circuits, the test cylinder operates autonomously from the propelling cylinders. Fed by an Atlas Copco air compressor, the intake air pressure of the test cylinder can be boosted up to 5 bar. The fresh air mass flow is measured with a Micro Motion Coriolis mass flow meter. Non-firing cylinders 2 and 3 function as EGR pumps (see Figure 1, the schematic layout of the setup). Their purpose is to generate adequate EGR flow, even at elevated charge pressures.

Fueling of cylinder 1 is provided by a double-acting air-driven Resato HPU200-625-2 pump, which can deliver a fuel pressure up to 4200 bar. An accumulator is placed near (~0.2 m) the fuel injector to mimic the volume of a typical common rail and dampen pressure fluctuations originating from the pump. The fuel mass flow is measured with a Micro Motion mass flow meter.

For measuring gaseous exhaust emissions, a Horiba Mexa 7100 DEGR emission measurement system is used. Exhaust smoke level (in Filter Smoke Number or FSN units) is measured using an AVL 415 smoke meter three times per operating point, of which the average value is logged. In this paper, we will refer to smoke as soot. Although it is acknowledged that the two are not the same, it is assumed that the qualitative trends reported in this paper will hold also for soot nevertheless. The engine is equipped with all common engine sensors, such as intake and exhaust pressures and temperatures, and oil and water temperature. This quasi steady-state engine data, together with air, fuel and EGR flows, as well as the regulated gaseous emissions, are recorded at 20 Hz for a period of 40 seconds by means of an in-house data acquisition system (TUeDACs) [26]. The average of these measurements is taken as the value for the operating point under investigation.

Finally, a SMETEC Combi crank angle resolved data acquisition system [12] is used to record and process in-cylinder pressure (measured with an AVL GU12C uncooled pressure transducer), intake pressure, fuel pressure and temperature and injector current. All of these channels are logged with a resolution of 0.1 °CA for 50 consecutive cycles. From this data, the average and standard deviation of important combustion parameters, such as CA10, CA50 and IMEP, can be resolved online calculated online with the associated SMETEC software.

ENGINE OPERATING POINTS

All tests are performed at 1200 rpm, with a fuel injection pressure and temperature set to 1500 bar and 30 °C, respectively. As shown in Table 5, the operating points are based on two intake pressure points with two mixture strengths. Finally, injection timings (SOI), separated by 5 °CA intervals, are chosen.

Table 5. Operating points

<table>
<thead>
<tr>
<th>Intake pressure [bar]</th>
<th>1</th>
<th>1.5</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aim load (IMEP) [bar]</td>
<td>7.5</td>
<td>12</td>
</tr>
<tr>
<td>EGR [%]</td>
<td>0</td>
<td>30</td>
</tr>
<tr>
<td>Aim λ [-]</td>
<td>1.45</td>
<td>1.00</td>
</tr>
<tr>
<td>SOI [deg CA aTDC]</td>
<td>-5</td>
<td>-5</td>
</tr>
<tr>
<td></td>
<td>-10</td>
<td>-10</td>
</tr>
</tbody>
</table>

STOICHIOMETRIC RATIO CALIBRATION

Because the compositions of the blends differ from normal diesel, the stoichiometric ratio of each blend should be calibrated accordingly. The stoichiometric ratio of EN590 diesel is roughly 14.3. The air composition is supposed to
contain 20.95% O₂ and 78.09% N₂, so that \([\text{N}_2]/[\text{O}_2] = 3.773\), when other trace gases are neglected. If the hydrocarbon is described as \(\text{C}_c\text{H}_h\text{O}_o\), the combustion reaction equation can be written as

\[
\text{C}_c\text{H}_h\text{O}_o + (c + h/4 - o/2)(\text{O}_2 + 3.773\text{N}_2) = c\cdot\text{CO}_2 + h/2\cdot\text{H}_2\text{O} + 3.773(c + h/4 - o/2)\text{N}_2
\]

The stoichiometric ratio can then be written as

\[
(34.41 \cdot (4c + h - 2o))/ (12c + h + 16o)
\]

Furthermore, constants \(c\), \(h\) and \(o\) of each blend can be calculated according to the volume fraction of the compositions, resulting in the stoichiometric ratios listed in Table 6.

### RESULTS AND DISCUSSION

#### SOOT AND NOX EMISSIONS

Generally speaking, soot in DI diesel engines is formed in the hot, fuel-rich core of the various jet-like burning diffusion flames. As shown in Figure 2, there is an overlap between the injection and the combustion event for all fuels, even at the earliest SOI. At later SOI values, more towards TDC, this overlap shrinks and therefore the role of diffusion combustion becomes less dominant.

As shown in Figures 3, 4, 5, 6, the soot emission of the saturated cyclic blend is not lower, even higher in some operation points than that of the corresponding aromatic blend. Moreover, when the SOI approaches TDC, this difference becomes larger. When combined, the above observations suggest that when the diffusion phase of the diesel combustion becomes relatively more important, the impact of fuel chemistry on soot is more profound. Considering that the combustion process, in particular the combustion timing, is a key factor for soot formation in diesel engine, further analysis of combustion phasing will be discussed in the next section.

The use of EGR to reduce NOₓ emissions from diesel engines has been investigated extensively before, for instance by Ladommatos [27], who concluded that the most significant effect of EGR is the reduction of the oxygen flow rate to the engine. This leads to a reduced local flame temperature during combustion and, thus, a reduced rate of (thermal) NOₓ formation. As shown in Figure 6, NOₓ levels indeed become much lower when EGR is applied.

For each of the four operating points, all blends follow the same trend (as compared to each other) as a function of injection timing. Normally, for the four operation points investigated, the soot emission of diesel is expected to be higher than that of both oxygenates, which is confirmed by the graphs of soot emissions in the two operation points without EGR (Figures 3 and 4), and the operation points of low load with EGR in Figure 5. However, as shown in Figure 6, in the operation point of high load with EGR, the soot emission of the two oxygenates is higher than that of normal diesel. This might be explained by a lower combustion temperature and associated poorer soot oxidation for the two oxygenate blends. This possible explanation is supported by the lower NOₓ emissions seen for the oxygenated blends in this work point.

Noteworthy is also that the soot trend in the work points with EGR differs from that in the work points without EGR. Specifically, the lowest soot emission in the operation point without EGR is found for an SOI of 10 °CA BTDC or 5 °CA BTDC. With EGR, 5 °CA BTDC is the SOI with the highest soot. EGR thus seems to reverses the soot trend, but the
relative soot ranking amongst the three fuels remains unaffected. This suggests that aromaticity influences the soot emission, given that the fuel oxygen percentage in both oxygenates is kept constant.

Since there is generally a trade-off between soot and NO\textsubscript{x} in diesel engines, it is interesting to compare soot with NO\textsubscript{x} trends. As can be inferred from Figures 3, 4, 5, 6, the aromatic blend, containing 15% 2-phenyl ethanol, yields comparable trade-offs to the aliphatic cyclohexane ethanol blend. This suggests that hydrogenation does not appear to be a value-adding process step when producing fuel from lignin.

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**Figure 3. NO\textsubscript{x} vs. soot emission at 7.5 bar gross IMEP, no EGR, and lambda=1.45, for diesel (blue diamonds), 2-phenyl ethanol 15% (green circles), and cyclohexane ethanol 17.1% (red squares)**

**Figure 4. NO\textsubscript{x} vs. soot emission at 12 bar gross IMEP, no EGR, and lambda=1.60, for diesel (blue diamonds), 2-phenyl ethanol 15% (green circles), and cyclohexane ethanol 17.1% (red squares)**

**Figure 5. NO\textsubscript{x} vs. soot emission at 7.5 bar gross IMEP, 30 wt% EGR, and lambda=1.00, for diesel (blue diamonds), 2-phenyl ethanol 15% (green circles), and cyclohexane ethanol 17.1% (red squares)**

**Figure 6. NO\textsubscript{x} vs. soot emission at 12 bar gross IMEP, 30 wt% EGR, and lambda=1.15, for diesel (blue diamonds), 2-phenyl ethanol 15% (green circles), and cyclohexane ethanol 17.1% (red squares)**

---

**FUEL ECONOMY AND COMBUSTION PHASING**

Ideally, any improvement in the soot-NO\textsubscript{x} trade-off should be realized without sacrificing fuel economy. However, as can be seen in Figures 7, 8, 9, 10, fuel consumption for both oxygenated blends is higher than that of diesel fuel in all of four operation points, with the corresponding standard deviation of the ID over 50 engine cycles. In most of the measurement points, the deviations of the results are less than 0.5degCA (even smaller than 0.2degCA in most cases).
However, one should take into account that the heating value of any fuel will inherently decrease with fuel oxygen content. Compared to the base fuel, the studied oxygenated blends have a reduced gravimetric LHV of 2.7% and 1.9%, respectively (Table 3). This could be more or less expected given the fuel oxygen content in the blends of 2.33 wt.-%. Based on this decrease in LHV, penalties in ISFC could be expected in the order of 5-6 and 3-4 g/kWh for 2-phenyl ethanol and cyclohexane ethanol, respectively. As can be seen in figures 7, 8, 9, 10, 11, 12, 13, 14, this roughly explains the observed penalties in ISFC, suggesting that the thermal efficiency of combustion process is not significantly affected by the presence of the cyclic oxygenates at the studied concentrations.

The fuel consumption of the aromatic blend is slightly higher than (<1%) that of the saturated blends when the engine is running in an operating point without EGR. This could be explained by the 0.7% lower LHV for the aromatic blend (table 3). The order of the fuel consumption appears reversed when EGR is utilized, except for the points with unexpectedly higher fuel consumption in Figure 11 at relatively late SOI. There might have been a problem with the EGR flow rate control during the measurement (see appendix).

As shown in Figures 7, 8, 9, 10, 11, 12, 13, 14, the ignition delays of the two kinds of oxygenates blends and reference diesel fuel are quite similar at high loads, with or without EGR. However, when the engine is running at low loads, the difference in ignition delay becomes larger, especially at the points where injection timing is close to TDC when the engine running with EGR. This might be attributed to the aforementioned control problem of the EGR flow rate (see appendix), which did not change the general trends of the soot and NOx emission comparisons.
SUMMARY AND CONCLUSIONS

Two cyclic oxygenates, 2-phenyl ethanol and cyclohexane ethanol, which could be synthesized from lignin, have been blended to diesel fuel and used to investigate the impact of oxygenate aromaticity on the soot-NOx trade-off and fuel economy in a DAF heavy-duty diesel engine. The goal of this study is to investigate whether or not the expensive hydrogenation step of 2-phenyl ethanol to cyclohexane ethanol is worthwhile from an engine performance point of view.

Based on the results, a number of general conclusions may be drawn:

Compared to diesel and for a given fuel injection timing, a 1-3% higher ISFC is observed for the oxygenated blends. This penalty is roughly in line with the expected penalty of 2.33%, based on the fuel oxygen content of both oxygenated blends.

Compared to diesel and for a given fuel injection timing, a longer ignition delay can be observed for both oxygenated blends. This can be explained by their lower CN (Table 3).

The longer ignition delay, in turn, coincides and may be the cause of significantly lower soot emissions and slightly elevated NOx due to a more prominent premixed combustion.

By also adapting the SOI to account for the longer ignition delay of the oxygenated blends, a significant simultaneous reduction in both soot and NOx can be realized, especially in operating points without EGR and without an additional penalty in ISFC.

At an equal fuel oxygen content, the aromaticity of lignin derived (model) cyclic oxygenates does not appear to
significantly influence either the soot-NOx trade-off or fuel consumption. This conclusion is of importance, given that the hydrogenation of the aromatic compounds found in lignin is a costly process.

REFERENCES


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DEFINITIONS/ABBREVIATIONS

BTDC
before Top Dead Center

CA50
Time of 50% Accumulation Heat Release

CA50
Time of 50% Accumulation Heat Release

CN
Cetane Number

EGR
Exhaust Gas Recirculation

HHV
High Heating Value

IMEP
Indicated Mean Effective Pressure

ID
Ignition Delay=CA10-SOI

ISFC
Indicated Specific Fuel Consumption

LHV
Lower Heating Value

MON
Motor Octane Number

RON
Research Octane Number

SOI
Start of Injection

TDC
Top Dead Center
APPENDIX

According to Figure 9, there is a strange point of the ISFC and the corresponding ID at low load with EGR (in the red circle). However, this particular point was found to deviate because of a controlling problem of the EGR flow rate, as witnessed by the oxygen flow rate in the exhaust pipe as shown in Figure 11.

![Figure 11. Oxygen flow rate in exhaust, at 7.5 bar gross IMEP and 30 wt% EGR, for diesel (blue diamonds), 2-phenyl ethanol 15% (green circles), and cyclohexane ethanol 17.1% (red squares)](image-url)