Utilization of HVO Fuel Properties in a High Efficiency Combustion System - Impact of Soot Structure on the Diesel Particulate Filter Regeneration Behavior -

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ABSTRACT: This work represents a continuation of the earlier results published by authors on combustion and emission investigation of Hydrogenated Vegetable Oil on a High Efficiency Diesel Combustion System (SAE Paper:2013-01-1677). In present work the investigations are extended to analyze the impact of HVO fuel on characteristics of Particulate Matter emissions and regeneration behavior of Diesel Particulate Filter (DPF). The experiments were performed with pure HVO fuel, petroleum diesel and Biodiesel (RME/B100). Results suggest that activation energy of HVO soot oxidation decreased by ~ 6 KJ/mol as compared to soot from diesel fuel, leading to a decrease of DPF regeneration temperature by ~ 43 °C.

KEY WORDS: heat engine, compression ignition engine, particulate matter, diesel soot particles, diesel particulate filters, DPF regeneration, biofuels, hydrogenated vegetable oil [A1]

1. INTRODUCTION

The interest in diesel exhaust aftertreatment technologies is growing due to upcoming stringent EU and US emission legislation. Amongst the various control strategies, the use of diesel particulate filters is identified as most effective and viable solution to reduce the emissions of particulate matter. However, the trapped particulate matter (PM) needs to be oxidized from the Diesel Particulate Filter (DPF) to reduce the back pressure on the engine. The fuel utilized for the active regeneration results in an overall fuel consumption penalty. Previous researches have reported a regeneration related fuel penalty in the range of 2 - 7 % depending upon engine operating conditions and test cycle (1). The target temperature at DPF is the most important variable that can influence the rate of PM oxidation. The typical thermal regeneration of the diesel soot takes place at temperatures ~ 600 - 630 °C. With the reduction in the DPF target temperature, the rate of PM oxidation decreases, and hence, the duration of the regeneration gets prolonged, which may lead to increased fuel penalty. The magnitude of the regeneration related fuel penalty as well as the trade-off between regeneration time and regeneration target temperature is depicted in Figure 1. The fuel penalty associated with DPF regeneration leads to additional CO₂ emissions with no useful work output. Therefore, in order to optimize the regeneration related fuel penalty, it is necessary to understand the soot properties and phenomenon of the DPF regeneration.

Concurrently, the focus of research is shifting towards the development and usage of bio-fuels as alternatives for fossil fuels. These renewable biological fuels have the potential to permanently reduce greenhouse gas emissions and as well as engine out soot emissions. The production of bio-based diesel fuels from vegetable oils is commonly accomplished using a process known as Trans-esterification. The product of Trans-esterification is Fatty Acid Methyl Ester (FAME), commonly

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known as “Biodiesel”. The utilization of Biodiesel (FAME) in higher proportions / blends may cause some operational and durability issues in the modern engines. Owing to this reason, the FIE (Fuel Injection Equipment) manufacturers have recommended to restrict the use of FAME blending up to 7% v/v (3-7). Furthermore, in the recent past (Oct 2012), EU Commission has issued guidelines to restrict the use of biodiesel (FAME), produced from food based raw materials, to maximum 5% v/v (8). In the current scenario, to full-fill the EU renewable energy targets in transporation, the Hydrogenated Vegetable Oil (HVO) looks to be a viable solution. The hydrotreating of vegetable oils (HVO) and animal fats is a process based on oil refining know-how and is used for the production of biofuels for diesel engines. In the process, hydrogen is used to remove oxygen from the triglyceride vegetable oil molecules and to split the triglyceride into three separate chains thus creating hydrocarbons which are similar to existing high cetane diesel fuel components. This allows the blending in any desired ratio to meet relevant diesel fuel standard and without any concerns regarding quality (9). Also commercial use as 100 % biofuel is demonstrated on several markets like USA, Austria and Nordic countries (10).

The characteristics of the emitted soot, is a function of in-cylinder formation conditions, where the thermal decomposition chemistry of the fuel plays a significant role (11-13). Thus, it is very essential to understand the impact of these new biofuel candidates on the soot characteristics and its consequences on the DPF behavior. In order to help realize these goals, a number of tools have been employed to investigate the PM emissions, composition, microstructure and its combustion behavior in DPF.

2. IMPACT OF BIOFUELS ON PARTICULATE MATTER AND DPF BEHAVIOR– A BRIEF REVIEW

In the earlier publication from this research group, authors have demonstrated that the utilization of HVO in a high efficiency combustion system results in a significant reduction of engine out carbon monoxide (CO), total hydrocarbons (THC), soot and engine noise (Combustion Sound Level) as compared to petroleum based diesel fuel (14). Concerning the particulate emissions, at low load operation, the use of FAME fuel (RME-B100) resulted in the increase of total engine out PM due to higher share of the SOF (Soluble Organic Frations), however at medium and higher load points, due to low SOF, the potential of soot reduction improves with FAME. The decrease of engine out PM with FAME is mainly due to its in-built oxygen in fuel molecular structure (14-16). On the other hand, the benefits in PM emissions with HVO are mainly attributed to its aromatic free composition as well as its lower distillation range as compared to the petroleum diesel (14). Previous studies have reported significant variations in the structure and oxidative reactivity of the soot derived from different petroleum based diesel fuels and biofuels (11-13,17-23). Mikkonen et. al have demonstrated that due to the reduced engine out soot emissions, the HVO fuelling resulted in a slower increase of exhaust gas back pressure in DPF, which will eventually lead to enhanced regeneration intervals and a significant reduction of regeneration related fuel penalty (24). In another study, increased regeneration interval with biodiesel (FAME) fuels was attributed to the increased share of SOF (Soluble Organic Matter) and volatiles with biodiesel, these additional SOF / volatiles provide reactive hydrocarbons to oxidize catalytically in the DPF (17). A study by Yamane et. al. have reported that the metal traces (residues of the trans-esterification) in the soot from biodiesel fuel, acts as active centers to enhance the soot oxidation rates, thereby, leading to self regeneration effect, which resulted in the prolonged active regeneration intervals with biodiesel/FAME (25).

Tomasek et. al., in their study of soot structure, by using High Resolution Transmission Electron Microscope (HRTEM) technique concluded that the structure formed was attributed to engine operating conditions and the fuel identity (18). Boehman and co-workers have compared the microstructure of soot using HRTEM and XRD (X-Ray Diffraction). Authors concluded that the HRTEM provide localised information about the base structural units of the crystals, whereas the XRD analysis was representative to an average value of crystals in the soot sample (19).

3. METHODOLOGY AND APPROACH

The primary aim of this work is to understand the impact of different generations of fuel candidates on oxidative reactivity of soot and its combustion behaviour inside Diesel Particulate Filters (DPF). Figure 2 depicts the methodology and approach adopted for the present research. The soot samples were collected from a High Efficiency diesel Combustion System (HECS), exceeding the requirements set for EU 6 emission limits for passenger cars (14, 26). These soot loaded DPF samples were subjected to a Temperature Programmed Oxidation (TPO) in an innovative Laboratory Gas Test Bench (LGB), to determine the kinetic parameters of the soot oxidation behavior. Furthermore, the soot extracted from DPF was used for microstructure studies using X-Ray Diffraction (XRD) technique. The fuels, engine, and sample acquisition procedures are described in following section.
3.1. Research Engine and Soot Sampling Conditions

The single-cylinder diesel research engine (HECS diesel engine) used for the present work is optimized for improved combustion characteristics with regard to low emission and high fuel efficiency. Reducing compression ratio, using a higher maximum cylinder and rail pressure, as well as improved EGR cooling resulted in lowest engine emissions possible to meet Euro 6 engine out NO\textsubscript{x} targets without any DeNO\textsubscript{x} aftertreatment, however, the application of a DPF is necessary to meet the PM mass and number limits\textsuperscript{(14, 26)}. The main specifications of the research engine are summarized in Table 1.

Table 1 Hardware specifications for the research engine

| Unit       | Specification  |
|------------|----------------|
| Swept volume | [lit.]          |
| Stroke x Bore | [mm] 88.3 x 75 |
| Valves per cylinder | [-] 4 |
| Compression ratio | [-] 15:1 |
| Max. cylinder pressure | [bar] 220 |
| Intake air pressure | [-] Max. 3.8 bar abs. |
| Max. injection pressure | [bar] 2000 |
| Nozzle hole number | [-] 8 (cone angle 153°) |
| Nozzle diameter | [mm] 0.109 (HFR 310) |

The experiments for particulate measurements were conducted at part load operation point, can be represented in New European Driving Cycle (NEDC) as well as in the future proposed World Harmonized Test Cycle (WHTC), depending on the vehicle inertia class. The specifications of the engine calibration parameters are summarized in the Table 2.

Table 2 Engine Calibration parameters

| Unit       | Specification  |
|------------|----------------|
| Engine speed | min\textsuperscript{-1} 2400 |
| Engine Load (IMEP) | bar 14.8 |
| Center of Combustion “\(\alpha_{50}\)” | \(^\circ\text{CA} \) at TDC 10.8 |
| Rail pressure | bar 1800 |
| Intake air pressure | bar 2.60 |
| Charge air temperature | \(^\circ\text{C} \) 45 |
| Exhaust gas back pressure | bar 2.80 |
| EGR Rate | % 35±2 |

Figure 3 depicts a schematic of DPF soot loading in engine test bench. The samples were collected downstream of a DOC to preserve the fluffy structure of the soot\textsuperscript{(27)}. Besides the particulate sampling for different physico-chemical analysis, the engine exhaust was monitored by exhaust gas analyzer for CO (infrared gas analyzer from Rosemount - NGA platform 2000 series), HC (Flame ionization detector, Rosemount - NGA 2000 series) and NO\textsubscript{x} (Chemiluminescence, EcoPhysics CLD 700 EL). Smoke emission was measured by an AVL 415S smoke meter and opacity was measured as laser pulse extinction at 530 nm.

3.2. Methodology of Soot Oxidation Kinetics

Kinetics of soot oxidation were studied on a Laboratory Gas Test Bench (LGB), using oxidants O\textsubscript{2} and NO\textsubscript{2} in representative exhaust gas mixture. In the LGB, it is possible to conduct the oxidation kinetics analysis by the independent control of key operating parameters such as temperature, space
velocity and exhaust composition. The reaction rate was measured close to realistic conditions and activation energy as well as frequency factor were evaluated. The product concentrations were measured using FTIR, (Fourier transformation infrared spectrometer), FID (flame ionization detector), CLD (Chemiluminescence) and NDIR (non dispersive infra-red) analyser. A simplified schematic layout of innovative Laboratory Gas Test Bench (LGB) is shown in Figure 4 (29).

\[ \text{Eq.1} \quad I_{\text{react}}(t) = \frac{V_{\text{soot}, \text{m3}}}{V_{p, \text{exh}, \text{m3/s}}} \]

With this premise, the LGB oxidant consumption was evaluated as first order reaction inside the soot volume \(^{(23)}\). To make a close approximation with the real DPF applications, soot loaded DPF samples were used for the analysis as depicted in Figure 5.

Fig. 5 A microscopic view of the soot loaded DPF segment and its specifications, used for microstructure (XRD) as well as oxidation kinetics analysis (LGB)

3.3. Test Fuels

Three different diesel fuels were used for the present investigation. The petroleum based diesel fuel was used as reference for baseline tests and other two fuels are derived from biogenic sources, out of which one fuel (i.e. FAME / RME also known as Biodiesel) has oxygen content in the fuel molecular structure. Another biomass derived fuel candidate selected for this study is a Hydrogenated Vegetable Oil (HVO). Figure 6 depicts the main fuel properties of the above mentioned fuels.

Fig. 6 Main fuel properties of the investigated fuels
4. TEST RESULTS AND ANALYSIS

4.1. Particulate Emissions

Figure 7 depicts the FSN and PM gravimetric emissions of the investigated fuels. The reduction in FSN with RME and HVO is ~ 78 % and ~ 50 % respectively. A similar trend was observed for DPF soot loading where a reduction of ~ 62 % and ~ 43 % was observed with RME and HVO fuels respectively. However the extent of soot reduction in both the measurements is not the same. It can be explained due to the fact that FSN measurements are mainly based on the degree of blackness of PM emissions, while PM mass measurement consider the organic fractions and volatiles as well.\(^{(23)}\)

![Figure 7](image)

FSN DPF PM Flow

| Fuel | FSN | PM Flow |
|------|-----|---------|
| B0   |    |         |
| RME  | 78% |         |
| HVO  | 50% |         |

Fig. 7  FSN (Filter Smoke Number) and PM mass flow from investigated fuels at stated load point (n = 2400 min\(^{-1}\); IMEP = 14.8 bar and ISNO\(\text{x}\) raw emissions maintained at Euro 6 level)

4.2. Microstructure Analysis using X-Ray Diffraction

The soot powder samples were tested in the Institute of Crystallography, RWTH University Aachen on Philips Typ Xpert PRO. The diffractometer uses the “Cu K\(\text{α}1\) (1.54051 Angstrom)” and “Cu K\(\text{α}2\) (1.54439 Angstrom)” radiations. The measurement and analysis of the intensity distribution and the subsequent derivation of parameters, in this given angle range, was carried out using X’pert High Score plus, Version 2.2b (2.2.2) (PANalytical). For the measurement of the samples, a Silicon (Si) single crystal holder was used. The advantage of this sample holder is that the Si single crystal is cut parallel to the (911)-lattice planes and there exist no reflections with Copper K\(\text{α}1,2\) ( Cu K\(\text{α}1,2\)) radiation in the 2 Theta range 0 ° to 180 ° and sample holder does not produce any diffuse background. Figure 8 shows the XRD pattern of the soot samples generated from different investigated fuels as well as reference graphite sample. It is very apparent from the curves that all the soot samples possess two significant characteristics peaks. The first peak at ~ 2\(\theta\) = 23° corresponds to 002 planes and the second peak at ~ 2\(\theta\) = 44° corresponds mainly to the 100 plane.\(^{(21)}\)

The analysis of these parameters is focused on the inter-planar spacing (i.e. \(d_{002}\) distance) and the size of the crystallites at graphitic sites within the soot particle. These crystallites have to be connected by some amorphous glue built by the aliphatic polymerized hydrocarbons. This glue might be responsible for the variation in the XRD pattern of engine soot samples when compared to the reference graphite. This impact is clearly visible in the base of the curves from graphite and engine soot samples. This suggests that carbon structures in the soot vary from highly structured graphitic sites to the highly disordered amorphous hydrocarbons. To regard the impact of background generated by amorphous hydrocarbons, in addition to the evaluation of the lattice spacings and of crystallite size using Scherrer formula, a new parameter named as “Structure to Amorphous Ratio“ abbreviated as \(\text{StA}\) was evaluated.\(^{(21)}\)

\[
\text{StA ratio} = \frac{A_s}{A_a}
\]

Where \(A_s\) is the indication of the area of peak under structured hydrocarbons and \(A_a\) is indication of the area of base under amorphous hydrocarbons as depicted in the Figure 8.

The main results derived from XRD analysis are summarized in Table 3. The carbon black was investigated as a model soot for generating baseline reference. As expected all the soot samples showed \(d_{002}\) length values higher than that of graphite (0.3354 nm)\(^{(28)}\) probably because of the fact that the graphene layers in engine soot samples are not totally planar as in graphite due to small hydrogen content (at the end, the round
shaped shell core structure requires a small hydrogen content. The typical range of the evaluated lattice parameters correspond well with the previous studies using engine soot samples \cite{19,20}. The smaller value of the number of graphitic layers per crystallite along c-axis (i.e. $L_c$/$d_{002}$) should indicate higher reactivity for B0 soot, however, in the present analysis; it does not follow the expected trend, as compared with the oxidation behavior discussed in next section. These results correspond well with Song \cite{20}, where the initial soot microstructure analysis using XRD, depicted a smaller stacking order i.e. $L_c$/$d_{002}$ ratio and crystallite width i.e. $L_a$ values for particles from petroleum diesel fuel compared to biodiesel and its blends.

Table 3 The crystal lattice parameters calculated from XRD analysis (The $d_{002}$ values of the graphite are ~ 0.3354 nm)

| Soot Type           | $d_{002}$ [nm] | $L_a$ [nm] | $L_c$ [nm] | $L_c$/$d_{002}$ [-] | C. Atom* [#] | StA Ratio [-] |
|---------------------|----------------|-----------|-----------|---------------------|--------------|--------------|
| Carbon Blackt (Ref. sample) | 0.361 | 4.39     | 1.58     | 4.38               | 2674         | -            |
| B0 Soot             | 0.364 | 3.82     | 1.2      | 3.30               | 1547         | 0.61         |
| RME Soot            | 0.360 | 3.64     | 1.48     | 4.13               | 1771         | 0.60         |
| HVO Soot            | 0.362 | 4.18     | 1.30     | 3.60               | 1946         | 0.44         |

* C-Atoms: # of C-atoms per crystallite calculated from basic knowledge about grid of C-atoms in graphite structure (Planar C-C-distance of 0.142 nm) and using crystallographic parameters, $L_a$ and $L_c$.

The StA ratio (Structure to Amorphous ratio) of HVO is lowest amongst all the fuels, which indicates a high share of the amorphous hydrocarbons in the soot composition compared to the share of the structured hydrocarbons. Finally, the dependency of these microstructure parameters will be compared with the oxidation behavior of soot samples as analyzed in the following section (refer to Chapter 5).

4.3 Analysis of Oxidation Kinetic Parameters

Figure 9 illustrates the Arrhenius plot of reaction rate constant, ln (k) with respect to temperature (1/T) for B0 soot, RME soot and HVO soot, completed in two phases, periodically with $O_2$ and/or ($O_2$+$NO_2$) as main oxidants. The activation energy ($E_a$) of the soot oxidation can be derived from the slope of the fitted Arrhenius curves. The results indicate that the activation energy of HVO soot oxidation for Soot/$O_2$ reaction decreases ~ 6 kJ/mol as compared to B0 soot. For the constant rate of reaction (k), at temperature ($T_{ign}O_2$) = 600 °C for B0 soot, the corresponding temperature for HVO soot comes out to be 557 °C. This implies that thermal oxidation temperature for HVO soot is shifted to ~ 43 °C lower than B0 soot.

Due to the increasing interest in the passive regeneration / CRT® (Continuously Regenerating Trap) behavior, the results of Soot/$NO_2$ oxidation from Arrhenius plot are interpreted for the investigated soot samples.

Table 4 Soot oxidation results from LGB experiments

| Parameter                  | B0 Soot | RME Soot | HVO Soot |
|----------------------------|---------|----------|----------|
| Activation Energy (KJ/mol) | Soot/$O_2$ ($E_a$,$O_2$) | 116 | 113 | 110 |
|                           | Soot/$NO_2$ ($E_a$,$NO_2$) | 60 | 58 | 56 |
| Regeneration Temperature (°C) | Active Regn ($T_{active regn}$) | 600 | 580 | 557 |
|                           | Passive Regn. ($T_{passive regn}$) | 300 | 278 | 263 |

The activation energy ($E_a$,$NO_2$) for B0 soot from the Arrhenius plot is calculated as 60 kJ/mol. The results of this work are consistent with other studies showing significantly lower activation energy with $NO_2$ oxidation as compared to $O_2$ oxidation \cite{12}. Similar to the the $soot/O_2$ oxidation, the temperature ($T_{ign}NO_2$) for Soot / $NO_2$ reaction is also reduced by ~ 37 °C for HVO soot as compared to B0 soot. The main results of the soot oxidation kinetic parameters are summarized in the Table 4.
5. SUMMARY OF RESULTS

The primary aim of present investigation was to evaluate the impact of HVO fuel on the soot characteristics and its consequences on the DPF soot oxidation behavior:

- The result indicates that the engine out soot emissions are reduced significantly with using biofuels. The HVO fuelling resulted in ~ 50 % reduction in smoke emissions and ~ 43 % reduction in gravimetric PM flow while the reduction with RME was ~78 % and ~ 62 % respectively.
- In order to examine the initial microstructure of the soot, the soot samples collected in DPF were subjected to X-Ray Diffraction (XRD) analysis. The lower values of crystallite width (Lc) as well as stacking order (Lc/d002) indicates smaller crystallite size, and hence, should lead to higher reactivity, however, the behavior of B0 soot is observed anomalous. A similar trend was also observed by Song (20). For the initial soot nanostructure studies using diesel and biofuel soot samples, the diesel soot sample, inspite of lower oxidation rate found to possess lower Lc and Lc/d002 values. Therefore, it suggests that, beside the size of the graphitic crystallites in soot there should be other influences on the oxidative reactivity.
- The StA ratio (Structure to amorphous ratio) derived from the XRD pattern, indicates the “Ratio of Structured Hydrocarbons to Amorphous Hydrocarbons” in the soot. The “StA ratio” of B0 and RME soot is quite similar, while, the “StA ratio” of HVO soot is lowest, indicating highest disorder (more amorphous structure) in the microstructure of HVO soot.
- The results of soot oxidation analysis from LGB experiments correspond well with the “StA ratio” derived using XRD, indicating a higher reactivity of the soot from HVO fuel as compared to the RME and B0. Due to lower activation energy, the thermal regeneration temperature and passive regeneration temperature of soot from HVO was reduced by ~ 43 °C and ~ 37 °C respectively, compared to B0 soot.
- The RME soot inspite of similar “StA ratio” than B0 soot, seems to oxidize at lower temperature than B0 soot. This suggests that in addition to the microstructure, there are other influences such as metal content and H/C ratio etc. which could influence the soot oxidation behavior as well.

6. KEY CONCLUSIONS AND FUTURE SCOPE OF WORK

The results of the “stated engine operation point” using present investigation methods suggest a considerable advantage in the DPF regeneration behavior with use of HVO fuel. It is well evident that the regeneration behavior of DPF depends to a significant extent on the properties of the trapped soot. The results of XRD analysis of the soot discussed in the current paper indicates that the oxidative reactivity of the soot can’t be explained solely, with the conventionally described crystal lattice parameters (such as Lc, Lc, Lc/d002). Therefore, the evaluation of StA ratio from XRD patterns, in this work provided a further insight to help interpret the oxidation behavior.

The overall results from present work suggest that the oxidation behavior of soot is not contributed by one parameter, rather, it’s a combined effect of several physico-chemical properties such as optical properties, micro-structure, metal content and elemental composition of soot (i.e H/C ratio, oxygen content). In order to better understand the relationship between the soot characteristics and DPF regeneration behavior, the results of proposed analysis methods will be published in the future publications.

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