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Factors affecting diesel fuel degradation using a bespoke, high pressure, fuel system rig

Authors:

Kesavan Gopalan[1,2], Christopher R Smith[3], Simon G Pickering[1], Christopher J Chuck[2], & Christopher D Bannister[1*]

AUTHOR ADDRESSES

[1]Department of Mechanical Engineering, University of Bath, UK

[2]Department of Chemical Engineering, University of Bath, UK

[3]BP Formulated Products Technology, Technology Centre, Whitchurch Hill, Pangbourne, UK

* Corresponding author - Dr. Christopher D Bannister (C.D.Bannister@bath.ac.uk)

Abstract

Recently, there has been automotive industry-wide impetus to reduce overall diesel vehicle emissions and fuel consumption by increasing fuel injection pressures within common rail systems. Many production fuel injection systems are now capable of delivering rail pressures of 1800-2000 bar with those able to achieve 3000 bar under development. In addition, there has been a gradual increase in the permitted FAME content in EN590 diesel from 5% to 7% with further increases to 10% proposed. With these changes there has been mounting speculation that increasing injection pressures, especially with elevated biodiesel content, could contribute to fuel degradation, deposit formation, fuel filter blocking and corresponding vehicle reliability issues.

In this investigation a bespoke, high pressure fuel injection rig was designed and commissioned to mimic conditions representative of those experienced within a modern vehicle engine. The impact of rail pressure, biodiesel content and accelerated testing conditions on the stability of diesel fuel and the deposit formation leading to filter blocking were assessed.

Despite the abundance of literature on lab-based biodiesel degradation, under these more realistic operating conditions it was found that biodiesel did not increase the likelihood of deposit formation within the high pressure fuel system, with the same level of filter blocking observed for B0 and B10 blends. This implies that the filter blocking problem caused by on board fuel degradation has the potential to occur broadly in a wide range of different fuels compositions. B10 fuel tested with a rail pressure of 2000 bar resulted in a pressure drop across the fuel filter of 0.5 bar within 12000 minutes (approximately 8.3 days), whilst the corresponding experiment at 1000 bar rail pressure showed no filter pressure increase. When using model (B10) fuel filter blocking was observed at both 2000 bar and 1000 bar rail pressure, however with the lower pressure at a much reduced rate, leading to the belief that the increases in rail pressures toward 2000 bar have a significant effect on the propensity of vehicle diesel filters to block. Measures taken to increase the severity of the test, such as recirculating injected fuel to simulate shear effects, were found to increase the rate of degradation but not change the chemical composition of the solids formed, thus implying that they were valid methods of reducing test durations without introducing new degradation mechanisms. The rig presented here is therefore a suitable accelerated testing system for assessing the behaviour of fuels under higher pressures that will be common throughout the global diesel fleet in the near future.

Nomenclature

ΔP	delta P (Pressure drop)
Bx	Biodiesel blend ratio (x=0, (i.e. B0) means no biodiesel, x=10 (i.e. 10) means 10 per cent biodiesel in diesel etc.)
Ca	Calcium
CHNO	Carbon, hydrogen, nitrogen, oxygen
cP	Centipoise
CR	Common Rail
CRP	Common Rail Pressure
cSt	Centistokes
FIE	Fuel Injection Equipment
FAME	Fatty Acid Methyl Ester
FT - IR	Fourier Transform Infrared Spectroscopy
FPS	Fuel Property Sensor
^{1}H NMR	Proton Nuclear Magnetic Resonance
HEFCE	Higher Education Funding Council for England
HEIF	Higher Education Innovation Fund
HP	High Pressure Pump
HPCR	High Pressure Common Rail
Hz	Hertz
IP	Induction Period
LP	Lift Pump
m	Multiplet (NMR peak)
Na	Sodium
OEM	Original Equipment Manufacturer
ppm	Parts Per Million
q	Quartet (NMR peak)
RME	Rapeseed Methyl Ester
s	Singlet (NMR peak)
SME	Soybean Methyl Ester
ULSD	Ultra-low Sulphur diesel
UOB	University of Bath

Introduction

Recently, tighter emission controls have led to major changes in both diesel fuels, such as the introduction of fatty acid methyl esters (FAME) and fuel injector technology. These factors have led to an increasing energy efficiency while simultaneously delivering reductions in CO and particulate matter. However, these changes, such as the higher injection pressures appear to have led to new types of fuel system deposits in the fuel delivery system.[1] This has also lead to quicker diesel injector 'failure' rates, manifested as a significant drop in engine performance. These injector internal deposits have an adverse effect on injection quality; signs of this include power loss, rough engine idling, startability issues and increased combustion noise.[1] This reduction in injector performance typically takes the form of changes to spray patterns and flow rates, which can affect the emissions produced by the engine, resulting in the vehicle no longer being compliant with environmental regulation.[2]

Although these fuel deposits can form throughout the fuel system (due to fuel recirculation), this insoluble matter will eventually deposit onto the fuel filters, though typically most interest has been paid to the impact on performance when located on or in the fuel injectors, filter blocking is a serious problem in its own right causing failure to start, vehicle breakdown and vehicles entering limp home mode due to fuel starvation. The deterioration of fuel and the formation of deposits are usually attributed to diesel fuel oxidation, with organic carbon being the predominant element though inorganic components have been found in the deposits also.[3]

Many factors appear to affect diesel deposits. For example, a large body of work has been published on the oxidative instability of FAME, and how elevated levels can lead to deposit formation, through radical formation of oligomers, which eventually crystallise out of the diesel blend.[4-6] Temperature is another key effect on deposit formation.[7] This is especially pertinent as recent developments in injector technology (such as the DENSO 2nd generation CRS) have seen injection pressures increase to 1800-2000 bar and the control over injection timing and quantity improve through the use of high response solenoid valves.[7] This higher injector pressure has shown to result in substantially increased fuel temperatures in the injection system.[8]

Several methods have been developed to monitor the oxidation stability of commercial diesel and biodiesel fuels. Most popular tests are based on the measurement of fuel properties after accelerated degradation under a standard defined condition. These tests include the measurement of the induction period (IP), acid number, kinematic viscosity, peroxides, and insolubles.[9, 10] In a recent study, measurement of the IP by a PetroOxy test was used to follow the oxidation kinetics and the formed oxidation products. They found that this technique required only a small amount of sample and a short analysis time allowing for fast and highly reproducible results. It also provided flexibility in the operating conditions, and offered the possibility to study fuel kinetic oxidation and perform successive oxidation runs on the same sample.[11]

While most FAME studies have been conducted in non-representative chemical laboratories,[12] some recent studies have assessed the impact of biodiesel blends directly on the fuel injection equipment (FIE).[13] For example, by heating the fuel, and through the injected fuel being returned to the fuel tank, rapid degradation was observed to occur.[14, 15] Fuel recirculation occurs in a vehicle under real-world conditions due to fuel return circuits (from the high pressure pump, rail and injector spill), the rig proposed by Omori *et al.* attempt to accelerate the degradation process by returning the fuel passing through the injectors to the tank rather than being consumed through combustion.[13] Questions have remained as to the validity of recirculating injected fuel within fuel systems rigs and whether this introduces additional degradation issues in testing not normally experienced in-vehicle. Rapid depressurisation within the fuel return circuit (inside the injector and pressure control valve) generates a large amount of heat. A study by Omori *et al.* has shown that fuel temperature can increase by up to 150 °C in 1000-2500 bar injection systems.[13]

In addition, recent developments in injection technologies appear to have contributed to an increased intolerance to fuel system deposits.[16] Previous work has demonstrated that increases in fuel system temperatures and pressures coincided with an increase in the number of reports of greater filter and injector deposits when vehicles were used with ultra-low sulphur diesel (ULSD) in the USA.[17] Studies have suggested that these are directly due to increased injector pressures and temperatures, as well as biodiesel content, that has caused solid formation which is depositing directly in the injector and filters.[3]

Two types of deposits have been recovered from FIE systems after extended running with FAME blends.[18] These are (i) white or yellow waxy appearance deposits and (ii) brownish lacquered appearance deposits. FAMEs containing unsaturated double bonds that are unstable and can degrade into a range of products including organic acids.[19] The reaction of these fatty acids with metal

ions (Na or Ca) can result in the former white / yellow waxy deposits.[2, 20, 21] The brownish lacquered deposits could also originate from fuel degradation products (i.e. polymerisation of aged FAMEs).[2] The oligomer substance generated by biodiesel oxidation deterioration causes deposits adhering to the inside of common rail system, resulting in adverse effects on performance. On a lab scale common rail system under accelerated test conditions, FAME oligomers were observed in the sliding sections of the injectors.[5, 22] Similarly, tests on common rail fuel pumps using pre-test highly oxidised B20 fuel have shown the formation of lacquered type deposits on the shaft bearing surface; this type of deposit is hard to remove and insoluble in oil, impacting on the useful lifespan of the pumps.[23] Another study by General Motors Company found that when severely oxidised B20 fuel was used, significant performance deterioration occurred. These issues were attributed to the formation of coagulated oxidised 'sludge' in the fuel lines and fuel tank (no analysis was performed on the constituents of this sludge) as well as this 'sludge', internal injector deposits were found and deposits had been formed on internal components in the fuel pump.[24]

A recent study by Barker *et al.* examined deposits found in injectors and fuel filters from a range of vehicles that operated with higher injection pressures. In this work, they demonstrated that a black (carbonaceous) polymeric organic solid deposited on the filter over time. These deposits were found to block the filter pores, reducing the number of pores available and thus reducing the performance of the fuel filters.[16] The deposits indicated possible precursor molecules, such as FAME and aromatic species from the parent diesel, which supported a complex fuel degradation mechanism involving more than just the biofuel content.

Building on this published body of work, in this study, a bespoke high-pressure common rail FIE rig was developed to be able to test the solid formation in diesel fuels effectively, and investigate the factors present in deposit formation on the fuel filter. Within this study, the validity of using factors such as fuel injection and fuel recirculation were examined to accelerate the testing, as well as the effect of injection pressure on solid formation, the injection event itself, the FAME content and online sensing of deposit formation.

Experimental Setup and Procedures

The FIE test rig was designed to be mimic as closely as possible real-world conditions, though deliver accelerated fuel degradation testing. To this end, the reaction parameters included variable injection pressure, an ability to heat the source fuel, control of the injection event and the ability to heat the injectors, through placement in a heating block (Figure 1, Table 1).
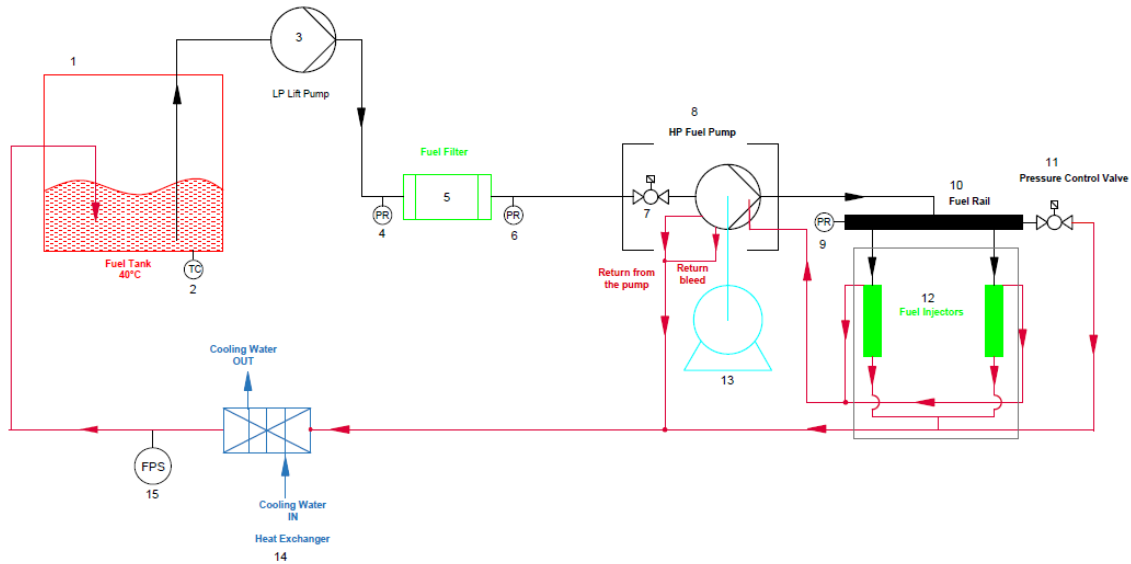
FIE Test Rig Instrumentation

Figure 1 - Simplified schematic representation of diesel fuel degradation rig.

Table 1 - Key for components labelled in Figure 1 above. All temperature measurements use K-Type thermocouples unless otherwise stated.

Label	Description	Label	Description
1	Fuel tank	9	Rail pressure sensor
2	Fuel tank temperature	10	Fuel Rail
3	Injection fuel lift pump	11	Rail pressure control valve (rail assembly)
4	Pre-Filter fuel pressure	12	Fuel injectors
5	Fuel filter	13	FIE pump drive motor (integrated in rig)
6	Post-Filter fuel pressure	14	Heat exchanger
7	Flow control valve (FIE pump assembly)	15	Fuel Property Sensor
8	HP fuel Common Rail pump		

The fuel rig was built incorporating a commercially available fuel injection system consisting of two solenoid diesel injectors, a common rail (CR), high pressure pump (HP), a lift pump (LP), a fuel filter, a fuel cooler and a fuel tank. A layout of the recirculation test rig designed for this study is shown in Figure 1. The rig also comprised an on–board fuel property sensor (FPS) which was used to measure physical properties such as dynamic viscosity, density, dielectric constant and temperature. The sensor was sourced from Measurement Specialties™ (Part no. FPS2820B12C4) and was designed to directly and continuously measure these properties. The sensor is based on a tuning fork technology and monitors the direct and dynamic relationship between multiple physical properties.

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The FIE pump was driven by an electric motor with an in-line torque flange recording the motor speed and torque. Spill returns from the common rail, high pressure pump and injectors returned to the tank via a heat exchanger to maintain the fuel at 40 ℃ in the tank. To accelerate the aging process, injected fuel could also be returned to the tank and recirculated.

The injectors were mounted in a custom-made aluminium block with integrated heater elements (cartridge heaters with internal temperature measurement) allowing the mounting temperatures to be set and controlled to simulate mounting within an actual engine cylinder head. The common rail pressure and injector actuation was controlled by a Stardex® common rail testing system which allowed selection of rail pressure up to 2000 bar and user-defined injection profiles (duration and frequency). Fuel temperature was measured at a number of points around the system in order to monitor the thermal cycle the fuel experienced. A control PC was used to operate the rig through which alarms and shutdowns could be defined.

Fuel selection and preparation

A batch of baseline diesel (B0, EN590) which contained only refinery additives and had not been treated with manufacturer-specific additive packs. In Western Europe, FAME is commonly produced from rapeseed oil (RME) and soybean oil (SME). SME is known to have lower oxidation stability than RME due to its higher proportion of polyunsaturated compounds and was therefore selected for its increased propensity to oxidation.[25] The baseline diesel was then used to blend a 10% v/v mixtures with SME. In order to ensure proper functioning of the diesel FIE, fuel properties must comply with the European specifications EN590 and can be blended with an EN14214 compliant FAME up to a maximum volume of 7%, but it is expected that this will increase to 10% in the coming years.[15] Therefore a 10% SME blend represents a high, but realistic future diesel/FAME mixture.

In order to simplify the chemical analysis and minimise the test time, a model fuel, incorporating the general chemistries found in diesel, was used. This model fuel was made from mixing individual compounds (an aromatic hydrocarbon, a FAME compound, a nitrogen containing heterocycle, a cyclic hydrocarbon and an aliphatic hydrocarbon.

Test Conditions

Prior to all tests, the rig was flushed repeatedly with the test fuel for a defined period of time. Reduced CR pressure and motor speed were used for the flushing cycle. In the fuel test rig used in this study, fuel was continuously recirculated and underwent a periodic thermal cycle which accelerated the degradation process. The fuel temperature inside the tank was regulated to 40 °C, chosen as a representative temperature of a standard vehicle. The tank was filled to two thirds of the capacity to allow air breathing while minimising system volume. The heating temperature of the two injector bodies was chosen based on the auto–ignition temperatures of the test fuels used and was consistent for all tests. The temperature was selected as a worst-case temperature of the cylinder head taking into account additional heat transfer generated by the combustion events. Each test was run continuously until a defined increase in the pressure drop (ΔP) across the filter was observed as ΔP was used as a measure of rate of solid deposition on the filter.

The matrix of experiments of this study is presented in Table 2. Test 1 was a baseline test with normal diesel fuel (B0) without FAME and keeping injector heating and CR pressure at 2000 bar. Test 2 was a repeat test of Test 1 to investigate the repeatability of the FIE rig. In Test 3, B0 was blended with 10% SME (FAME) and tested under same experimental conditions. In Test 4, fuel degradation was studied in the absence of the recirculated injection event to understand the effect of shear on fuel degradation. This was followed by studying the effect of pressure on fuel degradation in Test 5. Finally, in Tests 6 and 7, real-world fuels were replaced with simulated fuels to allow a deeper understanding of the effect of pressure on fuel degradation and deposit formation.

Table 2 - Matrix of test conditions.

Test #	Fuel type	Injection event ON / OFF	Rail pressure bar	FAME content % vol.
1	B0 (EN590)	ON	2000	0
2	B0 (EN590) repeat test	ON	2000	0
3	B10	ON	2000	10 (SME)
4	B10	OFF	2000	10 (SME)
5	B10	ON	1000	10 (SME)
6	Model fuel	ON	2000	10
7	Model fuel	ON	1000	10

Fuel sampling and Analysis

To give further information on the fuel degradation, fuel samples were taken periodically and the solids separated at the end of the test. Fuel samples were taken at the very beginning of the test (fresh samples), during the test (every 24 h) and at the end of the test (aged sample). Fuel properties such as the dynamic viscosity, dielectric constant and density were measured continuously using the on-board FPS sensor. Other analytical techniques such as elemental analysis (CHN), FT-IR and ^{1}H NMR were also used to examine fuel degradation throughout the testing.

The solid deposits from the filter were also analysed. The observed solid deposits were insoluble in most commonly used organic solvents such as hexane, ethanol, acetonitrile and diethyl ether. However, the majority could be dissolved in acetone. The extraction process involved the following steps;

 (1) Filter containing solids was soaked for 48 hours in acetone in a beaker (Figure 2a) to allow solids from the cellulose filter move in to the solvent phase (Figure 2b).

 (2) The acetone was removed *in vaccuo* from the liquor leaving behind a brown solid (Figure 2c).

 (3) The brown solid was subjected to hexane washes (5 X 20 ml) to ensure no trace fuel was present.

 (4) The resulting solid was washed with a small quantity of acetone (1ml), dried at 60 °C overnight and subsequently analysed.
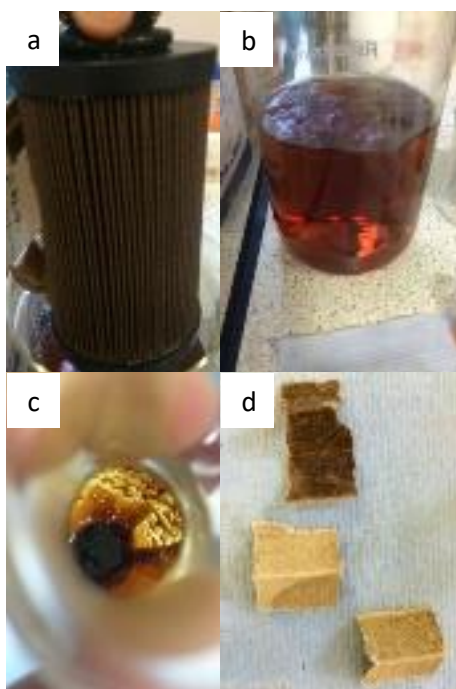
Figure 2 - (a) Aged filter containing solid. (b) Acetone liquor containing solids post filter soak. (c) Fuel insoluble solid residue obtained from filter. (d) Remaining dried filter.

Analytical Techniques

Kinematic viscosity

Kinematic viscosities were determined in accordance with ASTM D445 using a Canon-Fenske® capillary kinematic viscometer. Temperature modulation was achieved using a refrigeration unit. Samples within the viscometer were allowed to rest at the required temperature for a minimum of 5 minutes prior to viscosity measurement to allow temperature equilibration. The standard error was found to be lower than ±0.073 mm^{2}s^{-1} at -20 °C, ±0.128 mm^{2}s^{-1} at 20 °C and ±0.100 mm^{2}s^{-1} at 40 °C.

Elemental analysis (CHN)

Samples were tested by Medac Ltd.® using a FlashEA® 1112 Elemental Analyser configured as a CHN-O analyser for the determination of carbon, hydrogen, nitrogen and oxygen contained in the organic and inorganic chemical compounds and substances. The determination of CHN was performed in a single sample analysis, whereas oxygen was determined separately. The technique used by Medac® for the determination of CHN was based on the quantitative "dynamic flash combustion" method. The technique used to analyse oxygen was based on the Unterzaucher modified method.

Nuclear Magnetic Resonance Spectroscopy (NMR)

Proton NMR spectra were recorded using a Bruker® Advance III NMR spectrometer operating at 500.13 MHz for ^{1}H. Samples were analysed using ($(CH_3)_2CO$) at 25 °C using standard Bruker pulse sequences (TopSpin® 2.1). Typically ^{1}H spectra were acquired with a SW of 20 ppm, and 16 transients. Spectra were referenced using the residual solvent signal, at 2.05 ppm for ^{1}H.

Results and Discussion

Repeatability Evaluation

Evaluation of the experimental repeatability of the rig was undertaken by conducting repeat tests of the baseline B0 fuel under identical test conditions. The repeats were conducted under 'worst-case' conditions of 2000 bar rail pressure, injector heating active (190 °C) and recirculated injected fuel (Figure 3). The resolution of the pressure transducers in the pre and post fuel filter location was 0.01bar giving the appearance of discrete test points. At each ΔP the median time taken to achieve that pressure is shown, with error bars representing the maximum and minimum times at which that pressure was recorded. With fresh fuel and a new filter, there was an initial pressure drop across the filter of 0.01 bar. The error from these runs, calculated as the shortest time taken to achieve specific ΔP values, between the repeat tests demonstrated a variation in time of about 500 minutes seen at 0.1 bar ΔP (Figure 4). Although the increase in ΔP for the two repeat tests diverges above 0.07 bar, due to the nature of the experiment and the complexity of the fuel chemistry involved, the result is considered to represent good agreement. The magnitude of the variation should be kept in mind when considering results from perturbed parameter tests.
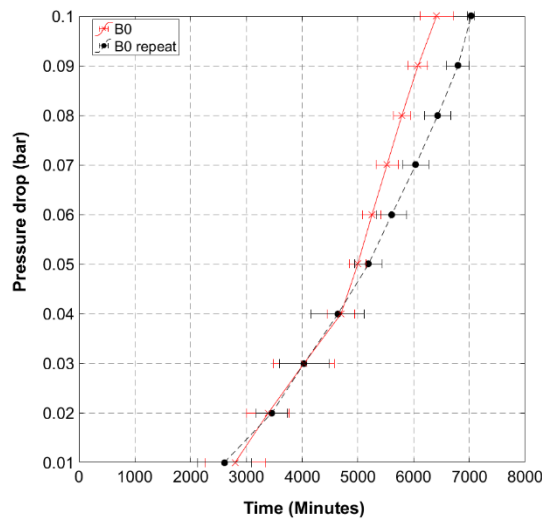

Figure 3 - B0 repeatability testing. Reaction conditions: Real-world fuel (B0), CRP: 2000 bar, Injection event: On, Heaters: On.
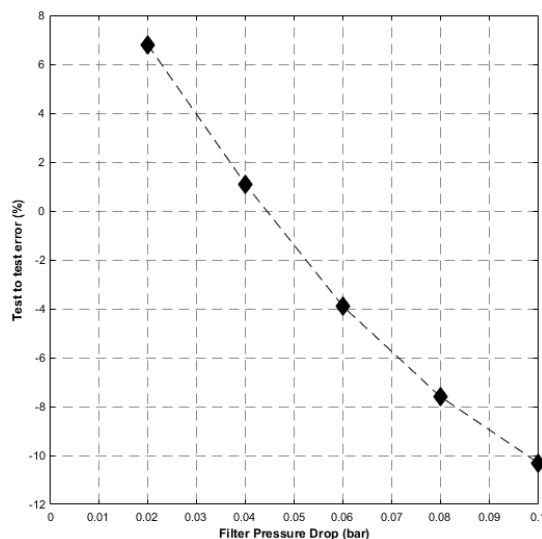

Figure 4 - B0 repeat test error variation with filter pressure drop.

Effect of FAME on fuel degradation

It has widely been demonstrated that the inclusion of FAME within EN590 diesel has led to a reduction in the stability of the blend. Numerous studies have examined the oxidation stability of FAME from various feedstocks concluding that increasing the degree of unsaturation leaves FAME susceptible to oxidation via a radical mechanism.[1, 14] Recent studies assessing the oxidation stability of diesel and biodiesel blends using a PetroOxy device (as per ASTM D7545 and EN16091) have concluded that the addition of 10% FAME (RME) reduced the induction period of the blend by 2.6 times (at 403K) compared with the baseline B0 diesel fuel indicating that FAME had a significant detrimental effect on the oxidation stability of the fuel.[11] The PetroOxy test exposes the fuel to elevated temperatures under a pressurised oxygen environment and, despite the findings related to the variations in stability of B0 and B10 blends, it is not clear if these differences would also be observed under real-world operating conditions within a vehicle fuel injection system.

The main direct impact of FAME oxidation on engine operability is not generally the change in the fuel properties but rather solid deposition. Therefore, the impact of FAME on fuel degradation and filter loading at 2000 bar with heated, active injectors was investigated. Figure 5 shows the increase in filter differential pressure with time for both B0 and B10 fuel. The addition of FAME to the diesel fuel did not result in the significant reduction in stability (characterised by a reduced induction period or rate of deposition) observed using the PetroOxy test and reported by Bacha *et al.*[11] While there are slight variations in the rate of ΔP increase between the two fuels between 6000 and 8000 minutes, the magnitude of the difference is small and within the bounds of experimental accuracy for the test as shown in Figure 3. Induction periods for both fuels lasted for approximately 2100 minutes with a very similar rate of subsequent deposition.

This works suggests that the formation of deposits under high and low pressures can be significantly different, therefore the use of more complex equipment and methods can supplement simple laboratory methods.
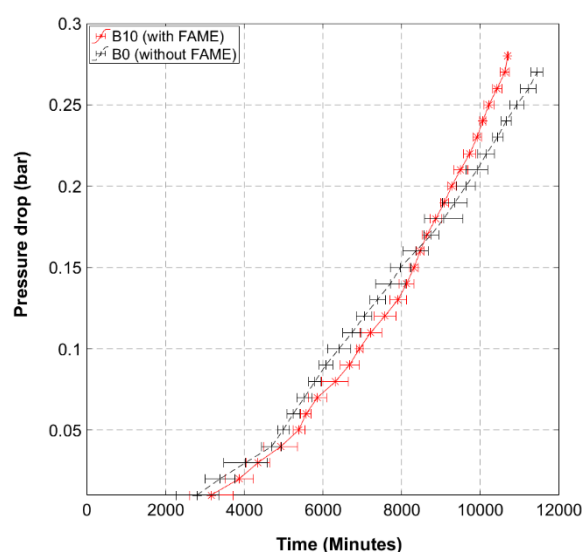

Figure 5 – Comparison of fuel degradation in the presence and absence of FAME – (Real-world fuels). Reaction conditions: Real-world fuel (B0 and B10), CRP: 2000 bar, Injection event: On, Heaters: On.

Injected fuel recirculation evaluation

As with previous injection rigs,[13, 14] the fuel degradation rig presented here, recirculated the injected fuel to accelerate the degradation process and reduce test times. It is possible that the cavitation and shear effects experienced by the fuel during injection are unique, compared to elsewhere in the vehicle fuel system and could give rise to different degradation processes than would be observed if the injected fuel was combusted and removed from the fuel system. For this reason, it was necessary to determine the impact of recirculating injected fuel on test durations and if, by utilising the injection event, the chemical composition of any resultant degradation products (primarily solid matter deposited on the filter material) remained consistent. To assess this effect, tests were undertaken using B10 fuel at 2000 bar rail pressures and the injector mountings maintained at 190 °C with and without an injection event. The system setup remained unchanged for both tests, with fuel supplied to the injectors

from the rail and returned via the spill line in the same way, with the only difference being that the injectors were not energised to initiate injection events. This was so that fuel passing through the heated, inactive, injectors to the spill line would still experience the temperature changes associated with the heated injector mounting (Figure 6).

The B10 fuel begins to undergo degradation after approximately 2900 minutes with the active injection event and, slightly later, at approximately 3900 minutes without. After the onset of the ΔP rise, the rate at which deposition occurs is significantly higher with injection resulting in a 42% and 45% reduction in the time taken for the ΔP to reach 0.1 and 0.15 bar respectively. The variation in the ΔP between the two conditions (approximately 71% at 0.1 bar) is an order of magnitude greater than differences observed in repeatability tests (7% variation at 0.1 bar shown in Figure 3) and is thus considered to be significant.

^{1}H NMR of the acetone soluble solid residue was compared for both tests to determine any structural differences in the residue resulting from the injection event (Figure 7). It should be noted that the resin of the filter is also soluble in acetone, and is observed in both spectra. However, after extraction from the filter material, the ^{1}H NMR spectra are highly similar to one another, showing no significant differences in the organic material between each run. The elemental analysis of the solids was also assessed, after manually being removed from the filter, and demonstrate the same relative ratio of carbon, hydrogen, oxygen and nitrogen ratios within each of the solids (Table 3). The injection event therefore does not seem to substantively alter the composition of the solids formed, but successfully reduced the test duration by approximately 45%.

Either the shear and cavitation doesn't play a large role in this mechanism or it is possible that similar shear and cavitation events are being experienced elsewhere within the rig, albeit to a lesser degree. Possible locations for these events would be the rail pressure regulator valve (which maintains the desired fuel pressure within the common rail system) and the valve chamber within the injectors. In both locations the fuel is discharged through a small orifice while the pressure is rapidly reduced from 2000 bar to approximately atmospheric in a similar way to the injection process.
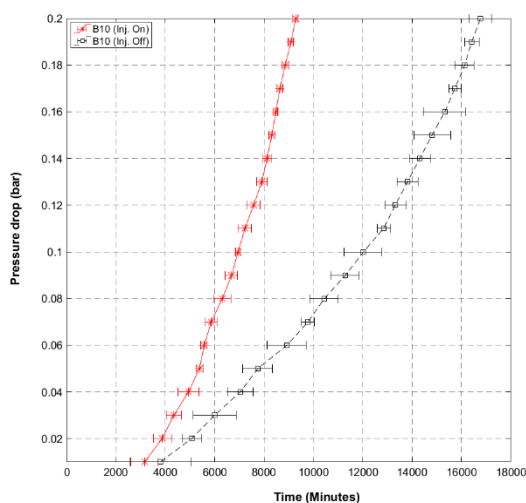

Figure 6 - Effect of shear. Reaction conditions: Real-world fuel (B10), CRP: 2000 bar, Injection event: On and Off, Heaters: On.
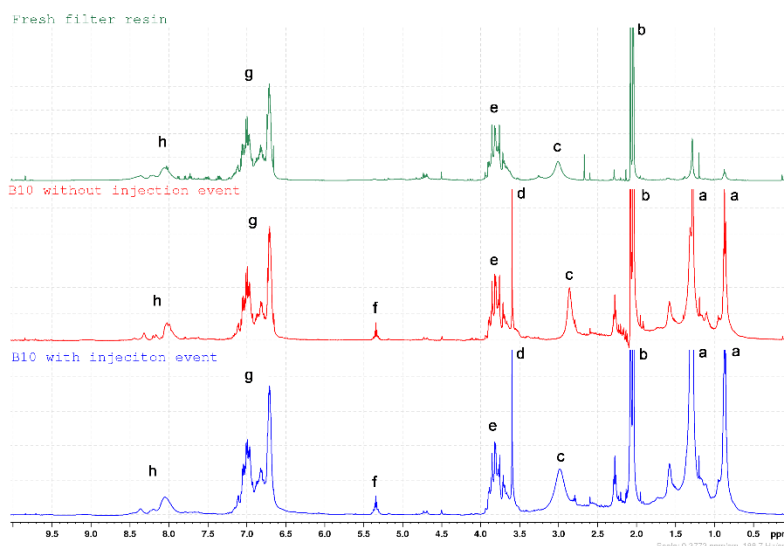
Figure 7 - NMR of Injector on and off solids. Peak assignment - a: (0.7-1.5 ppm), -CH$_2$, -CH$_3$; b: (2.04 ppm), solvent peak (deuterated acetone); c: (3 ppm, broad singlet) -OH peak; d: (3.6 ppm, singlet)-CH$_3$ ester peak corresponding FAME; e: (3.6-3.9ppm, multiplet) α-protons; f: (5.3-5.4 ppm, quartet) olefin peaks corresponding to FAME; g: (6.5-7.2, multiplet) aromatics; h: (8+ ppm) Heteroaromatics (nitrogen/oxygen containing aromatics).

Table 3 - Elemental analysis for B10 fuel with injection event on and off.

Test	% C	% H	% N	% O
B10 – Injection event ON	72.9	8.38	0.65	18.09
B10 – Injection event OFF	73.4	8.23	0.60	17.12
Fresh filter	55.10	9.81	0.18	14.58

Fuel property sensor evaluation

Within the degradation rig designed for this study, a fuel property sensor (Measurement Specialties™ FPS2820B) was installed in-line for continuous monitoring of temperature, dynamic viscosity, density and dielectric constant. The sensor operates based on the tuning fork technology whereby the frequency of oscillation will vary in relation to the viscosity of the fluid. It is important to assess the viscosity as the differential pressure across the fuel filter is also affected by changes in fluid properties as well as deposition and element blockage but, in conjunction with data from the FPS, could isolate the relative effects of the two contributing factors. For this reason the FPS was evaluated to assess its suitability for specifically measuring changes in fuel viscosity.

The FPS measured dynamic viscosity against time during the B10 degradation test remains broadly constant for the initial 3000 minutes before increasing rapidly (Figure 8a). This trend is broadly in line with the ΔP data presented for the injection ON test when using B10 Figure 6. During the test, samples of the fuel were collected at 24 hour intervals for chemical analysis and these samples were tested offline to determine the kinematic viscosity using a Cannon Fenske® viscometer (size 200, range 20-100 cSt). The FPS viscosity was plotted against the corresponding offline sample kinematic viscosity in order to verify the sensor measurements (Figure 8b). Despite a clear increase in FPS viscosity being observed over time, there is no correlation between the sensor and sample measurements. In addition, the FPS sensor recorded dynamic viscosity ranging from 2 cP to 12 cP (600 % increase) while analysis of the collected samples saw no significant change in kinematic viscosity which, instead, ranged from 3.09 to 3.18 m^{2}s^{-1} (2.9% increase).

Considering the FPS method of operation, which evaluates changes in resonant frequency of an integrated tuning fork, it is possible that the solids being formed during degradation were adhering to the sensor surface and altering the mass of the vibrating parts. An increase in mass would result in a decrease in the frequency of vibration and be interpreted by the sensor as an increase

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in viscosity. This theory was strengthened when it was observed that FPS viscosity measured at the beginning of repeat tests did not match the viscosity of the fuel at the start of the initial test, instead it recorded a reading which equated to the measurement at the end of the previous run (data not shown). To return the FPS to its initial state required the sensor to be soaked in acetone for a minimum of 1 hour to remove deposits.
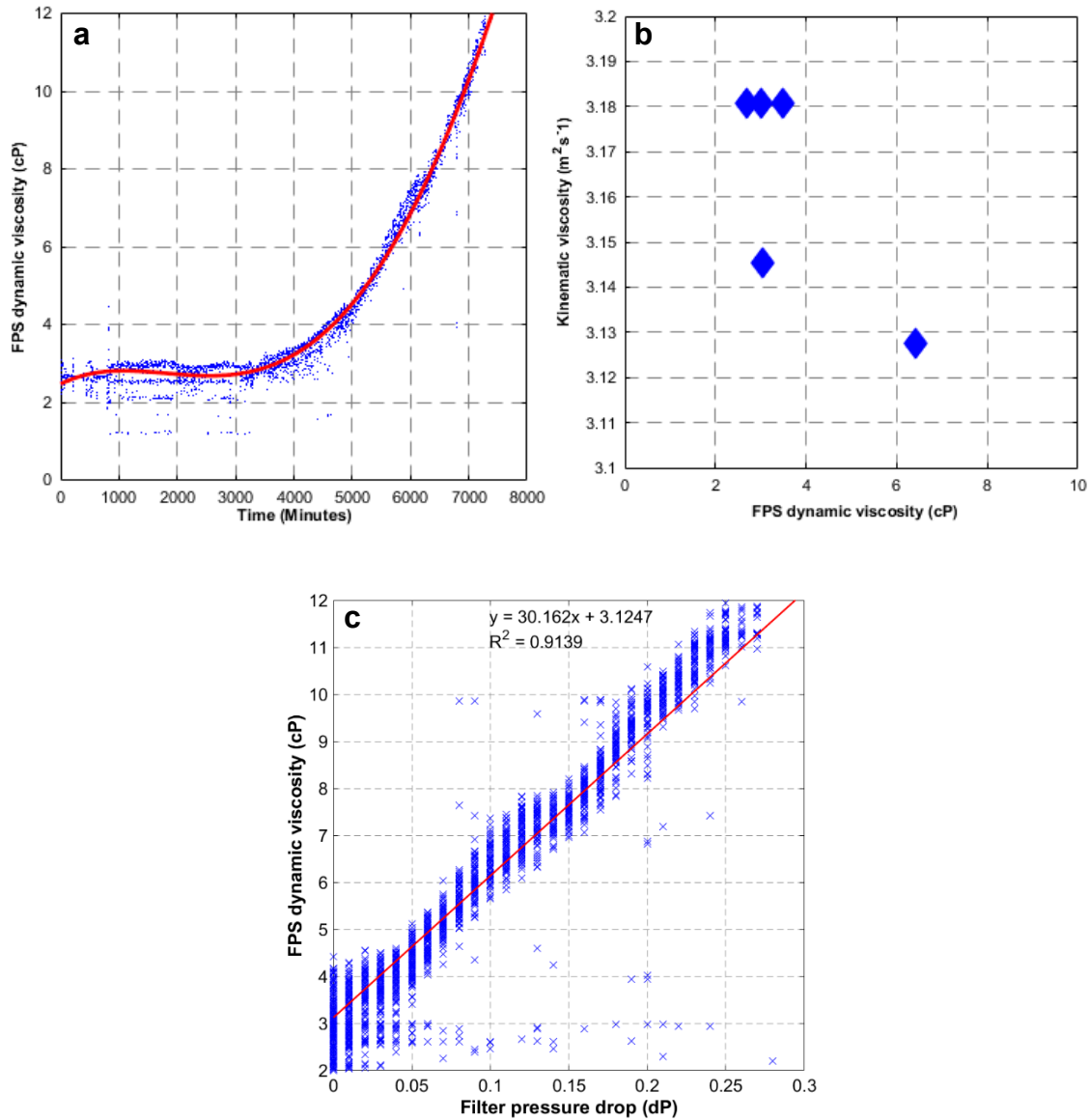

Figure 8 - FPS data from B10 degradation testing. A - Plot of sensor dynamic viscosity; B - comparison between FPS viscosity and corresponding offline fuel sample test; C - FPS viscosity against filter pressure differential (ΔP).

The FPS dynamic viscosity data was plotted against the measured filter ΔP and indicated a good correlation between the two factors (Figure 8c). This further suggests that the ability of a tuning fork-style FPS sensor to measure fuel viscosity is limited under conditions where degradation and solid formation are occurring at an accelerated rate and, instead, provides additional data on the rate of solid deposition within the FIE system. While this is a useful method of corroborating the filter differential pressure readings, and could potentially be used as a method of detecting solid formation, it does not, under these extreme conditions, provide useable information on the fluid properties themselves.

Effect of fuel injection pressure on solid deposition

A primary concern facing the fuel industry (and automotive OEM's), is the impact that increases in fuel rail pressures may have on the stability of diesel fuel and the subsequent production of solids leading to fuel filter blocking and vehicle reliability issues. The relationship between rail pressure and deposit formation was therefore examined. Tests were conducted using B10 fuel with the heated injector mounting and the injection events active. A comparison between filter pressure drop at 1000 bar and 2000 bar was undertaken with the tests run for a fixed duration of 10 days (Figure 9). No degradation was observed in the B10 fuel at a rail pressure of 1000 bar within the test duration (the step at 10,000 minutes is attributed to a stop and restart of the rig and not degradation as the ΔP does not continue to rise after this change), this was significantly different to the testing at 2000 bar, which showed a rapid increase in the measured filter pressure drop after approximately 5000 minutes. This highlights the significant detrimental impact that increasing rail pressure can have on diesel fuel stability.

As no filter deposition was observed at 1000 bar due to an increased induction period, no information could be gleaned into any potential variations in the rate of filter loading after the onset of degradation. To assess the rate of deposition over a shorter timeframe a synthetic fuel, composed of four model compounds chosen to mimic diesel (described in the experimental setup and procedures) was used to minimise the induction period and allow examination of the rate of deposition. The ΔP increase at 1000 and 2000 bar rail pressures when using the synthetic fuel are shown in Figure 10. Due to the change in fluid properties, with fresh fuel and a new filter, there was an initial pressure drop across the filter of 0.04 bar for this test. Using synthetic fuel reduced the time taken to observe an increase in ΔP at 2000 bar by approximately 70% compared to the B10 fuel previously tested and shown in Figure 9. At 1000 bar a rise in ΔP was observed after approximately 2100 minutes - a 40% increase compared to the 2000 bar condition. By the significant reduction in the induction period the subsequent rate of deposition can be evaluated. At 2000 bar the rate of deposition on the filter increases slightly with time apparent by the non-linear nature of the ΔP curve. At 1000 bar, the increase in ΔP is much reduced and far more gradual, with a near-linear deposition rate. This implies that increasing the rail pressure from 1000 to 2000 bar, not only reduces the induction period by an estimated 50%, but also significantly increased the subsequent rate of solid formation and filter loading. Applying these findings to the real world, increasing rail pressures would be expected to necessitate a reduced fuel filter service interval or re-sizing of the filter itself to account for the predicted increase in solid deposition.
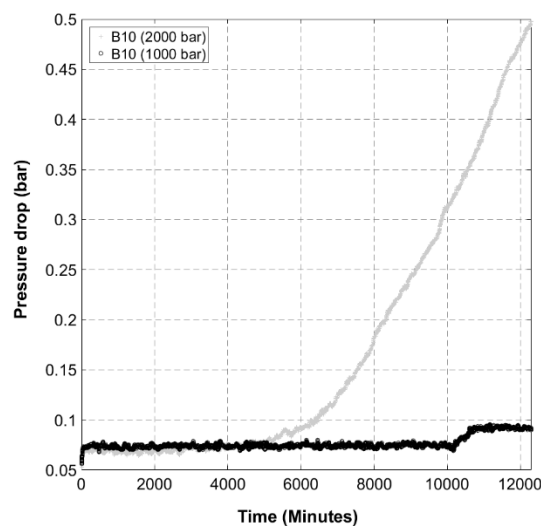

Figure 9 - Effect of pressures on diesel fuel degradation. Reaction conditions: Real-world fuel (B10), CRP: 1000 and 2000 bar, Injection event: On, Heaters: On.
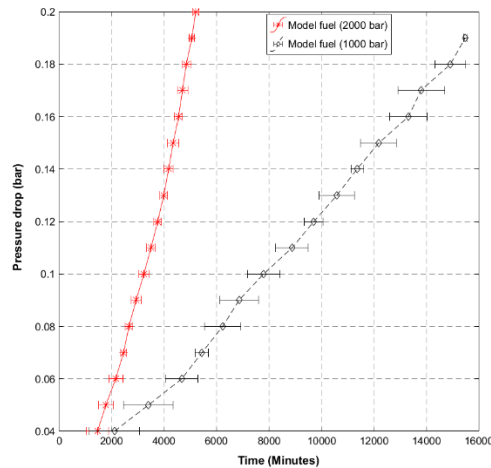
Figure 10 - Effect of pressure on synthetic fuels. Reaction conditions: Model fuel, CRP: 1000 and 2000 bar, Injection event: On, Heaters: On.

Conclusions

A bespoke fuel degradation rig was designed and commissioned which allowed the investigation of the impact of test conditions and fuel composition on deposit formation and filter loading. Contrary to the findings of standard oxidation stability tests, in particular the PetroOxy test, the addition of 10% v/v FAME was found to have no impact on the rate of filter deposition and associated pressure differential. Recirculating injected fuel within the rig was found to significantly reduce test durations (by approximately 45%) while not altering the composition of the deposits formed. Recirculation is therefore a valid method of accelerating tests without adversely affecting the validity of the investigation. In-line tuning fork-style fuel property sensors are susceptible to solid deposition within the fuel system and therefore do not, under these extreme conditions, provide useable information on the fluid properties. However, the sensors do give an indication of the rate of solid formation and the corresponding fuel filter loading. Using B10 diesel fuel, tested with a rail pressure of 2000 bar resulted in a pressure drop across the fuel filter of 0.5 bar within 12000 minutes, whilst the corresponding experiment at 1000 bar rail pressure showed no filter pressure increase within the timeframe of experiment (approximately 8.3 days). Using a model B10 fuel mixture, degradation was observed to occur at both 1000 and 2000 bar, but the rate of filter loading was reduced by approximately 50% at 1000 bar compared to 2000 bar. These findings suggest that vehicle filter blocking has the potential to occur broadly in a wide range of different fuels compositions. While lower rail pressures, typically seen in light duty diesel applications, can cause filter blocking, the propensity to block increases significantly with an increase in operating pressure.

Acknowledgements

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