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Diesel Injector Deposits – An Issue That Has Evolved with Engine Technology

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ABSTRACT

Diesel engines have traditionally been favoured in heavy-duty applications for their fuel economy, robustness, reliability and relative lack of fuel sensitivity. Recently it has seen a growth in its popularity in light duty applications due particularly to its fuel efficiency. However, as the engine technology and particularly the fuel injection equipment has evolved to meet ever stricter emissions legislation the engines have become more sensitive to deposit formation resulting from changes in fuel quality. This paper reviews bouts of concern over diesel fuel injector deposits, possible causes for the phenomenon and test methods designed to screen fuels to eliminate problems.

INTRODUCTION

It is now widely claimed that at the 1900 Exposition Universelle in Paris Rudolf Diesel exhibited a Diesel engine running on peanut oil thereby presaging the use of bio-fuels. Whilst it may be true that at the Paris expo a diesel engine ran on peanut oil it is far from true that Diesel designed his engine to run on vegetable oil.

In 1892 Rudolf Diesel first patented a process that was incorporated into internal combustion (IC) engine design to produce what has become known as the Diesel engine. In his patent Diesel envisioned that the process would operate with a wide variety of fuels; “Alle Brennmaterialien ih allen Aggregatzuständen sind für Durchführung des Verfarhens brauchbar” [1]. This roughly translates as “Every kind of fuel in any state of aggregation is suitable for carrying out the process.” However, the original patent goes to great lengths to exemplify a mechanism for delivering a controlled quantity of pulverised coal into the combustion chamber. This was obviously Diesel’s preferred choice of fuel, and with good reason, coal has a very high carbon content and hence a high calorific value. Vegetable oil on the other hand has a lower carbon content and lower calorific value. The first prototype engines were unsuccessful due to the failure of Diesel’s proposed method of delivering the pulverized coal into the combustion chamber. Since then there have been periodic bouts of renewed interest in running diesel engines on coal or coal slurries [2-8]. However, even recent work by Soloiu et al [9] using a charcoal slurry confirmed fuel injector fouling even after only one hour of engine operation.

When the first experimental engine ran in January 1894 it relied on liquid fuel but this was introduced using compressed air to atomize the fuel. In 1898 Diesel filed a patent entitled “Method of and Means for Regulating Internal Combustion Motors” [10]. This patent was in effect a patent covering the fuel injection equipment, or as Diesel termed it “The regulating appliance for effecting this admission of secondary combustible …” This design still relied on compressed air stored in a reservoir to force the fuel into the combustion chamber. However, Diesel continued to specifically include “solid, liquid, gaseous or vapour, or mixtures of these kinds,” as fuel sources. It was becoming clear that the new Diesel engine had great potential if the fuel injection system could be improved.

In 1910 the British engineer James McKechnie filed a patent [11] which outlined a system analogous to recent mechanical unit injectors. A fuel pump was used to pump fuel into the injection system where an engine driven cam and spring mechanism was used to generate the high pressure necessary to actually inject the liquid fuel directly into the combustion chamber. In McKechnie’s patent the pressures quoted are “from 2000 to 6000 lbs per square inch” i.e. 13.8 MPa to 41.4 MPa. Almost two years later McKechnie filed another patent outlining what today is known as the “common rail” system “in which the fuel under pressure passes into a main supply pipe from which it is supplied to the injection valve for each cylinder” [12]. Unfortunately, McKechnie did not disclose the “rail pressure” of his invention but it must be assumed that it was of the same order as mentioned in his earlier patent.

At around the same time Thomas Gaff, an American engineer proposed using a electrically operated solenoid to actuate the fuel injectors connected to a fuel accumulator [13]. However, in the first quarter of the twentieth century electronic engine management was still in the future and diesel engines were purely mechanical devices unlike the spark ignition engines which required electricity to produce the spark. The Gaff system was not therefore immediately adopted and it was over half a century before the common rail system with electronically actuated fuel injectors became the system of choice.

When Nippondenso Co Ltd patented their common rail system in the late 1980s [14], the rail pressure had risen to a "permissible pressure of 150 MPa" [15]. By the start of the twenty-first century people were proposing systems with pressures above 200 MPa [16, 17] and we now have systems capable of producing 300 MPa [18, 19].

It is not documented for how long Diesel's Paris expo engine ran on peanut oil; nor is it documented what effect this had on the rapidly evolving fuel injection equipment. However, work by Barsic and Humke [20] suggests that using vegetable oil will result in significant deposit formation on fuel injectors. An example of the deposit formation after approximately 20 hours of running on peanut oil is shown in Figure 1.

Figure 1. Deposit formation when running on peanut oil [20].

What is also unknown is what effect such deposits would have had on early diesel engines. What is known is that the Diesel engine and in particular the fuel injection equipment (FIE) have evolved to meet ever more stringent emissions legislation and customer requirements. Within this evolutionary environment there have been bouts of concern over the effect of deposit formation within the FIE and as a result there have been repeated attempts to define a test protocol to assess the propensity of different fuels to form deposits using market relevant engine technology of the time.

Whilst Diesel's early engines were of the direct injection (DI) design, many compression ignition engines of the first quarter of the twentieth century were of the indirect injection (IDI) type ; employing an ignition-chamber or ante-chamber into which the fuel was injected [21]. As the volume of this pre-chamber was obviously less than the total combustion chamber volume it meant that the mixture in this chamber was richer than the overall mixture strength and therefore more prone to soot formation. It can therefore be assumed that an IDI type Diesel engine would be more prone to symptoms of injector deposits. This supposition is to an extent confirmed by the fact that some of the first published work relating to diesel injector deposits was on IDI type engines. The following sections review some of the work that has been performed to assess the effects of deposit formation, the various test methods that have been used to asses fuel and fuel additive performance, current issues and research and finally draws some conclusions.

WHY WE MUST CONTROL INJECTOR DEPOSITS

As noted in the introduction there has always been a tendency for deposits to form in fuel injection systems and to an extent this has been allowed for in the design of the FIE. However, with the introduction of ever more stringent emissions regulations the variability in emissions as a result of this allowance has become more of an issue.

Due to legislation regarding permissible smoke emissions automotive diesel engines tend to be "smoke limited", i.e. the maximum fuelling rate is limited to prevent excessive soot formation and hence smoke emissions. Work in California by Irish and Mattson [22] showed a significant increase in smoke emissions throughout the operating range for "a popular six-cylinder, four-cycle naturally aspirated engine" after it had been operating in a truck for 32,000 miles (~51,500 km) of normal service. This is shown in Figure 2.

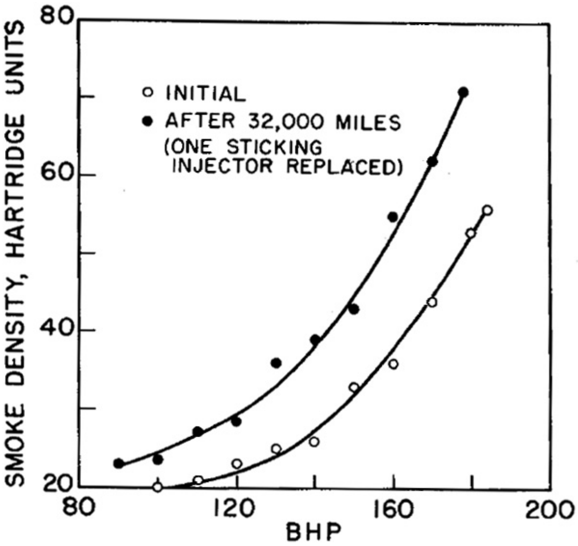

Figure 2. Change in smoke emissions due to nozzle fouling [22].

Although it is not explicitly stated, it is assumed that these engines were of the direct injection (DI) type. Irish and Mattson conclude "spray tip deposits must be a major cause of smoke and power loss …" and "… that tip deposits are harmful only when they disturb the spray pattern."

Montagne et al [23] illustrated visually the build-up of deposits on a pintle type fuel injector needle as used in an IDI type engine; this is shown in Figure 3. The work was performed using a combination of a single cylinder test bench engine and a production four cylinder engine. However, the cylinder configuration and FIE was the same for both engines; namely a Ricardo Comet V type pre-chamber, 93mm bore and 80mm stroke with a Bosch VE 4/9 injection pump and Bosch DNOSD 189 injector.

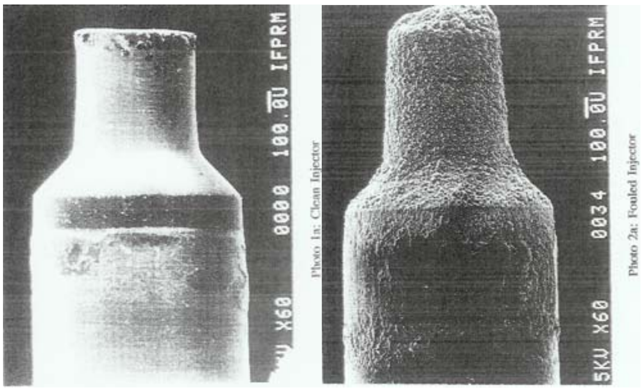

Figure 3. Clean and coked injector pintles [23]

This work was part of a programme to develop a method to assess the deposit forming potential of fuels and will be discussed later in this paper. However, this work also clearly demonstrated the effect of the deposits on the emissions performance of the engine. Figure 4 uses data taken from reference [23] and shows the effects on particulate matter (PM) emissions as a result of deposit build-up over 1000 km of driving. The PM emissions are expressed as g/test and were measured over a hot ECE 15 test cycle. The work performed by Montagne et al clearly demonstrated differences in behaviour between different fuels and particularly as a result of including fuel additives.

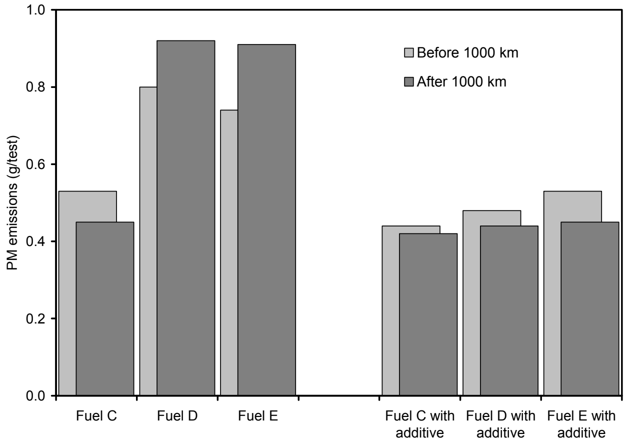

Figure 4. Data from [23] showing PM emissions

Reading et al [24] performed work to specifically to investigate the effects of fuel detergents on nozzle fouling and emissions. Again work was conducted on an IDI type diesel engine. For their work they used a vehicle with a 2.5 ltr, 4 cylinder, turbo-charged engine. Emissions measurements were made using the standard ECE 15 test sequence. A nozzle fouling index was determined by measuring the percentage fouling (i.e. loss of flow through the nozzle) at three different needle lifts and taking the average of these three values. The needle lifts used were 0.1 mm, 0.2 mm and 0.3 mm. The authors were then able to plot the emissions against the degree of nozzle fouling. Their results are shown in Figure 5.

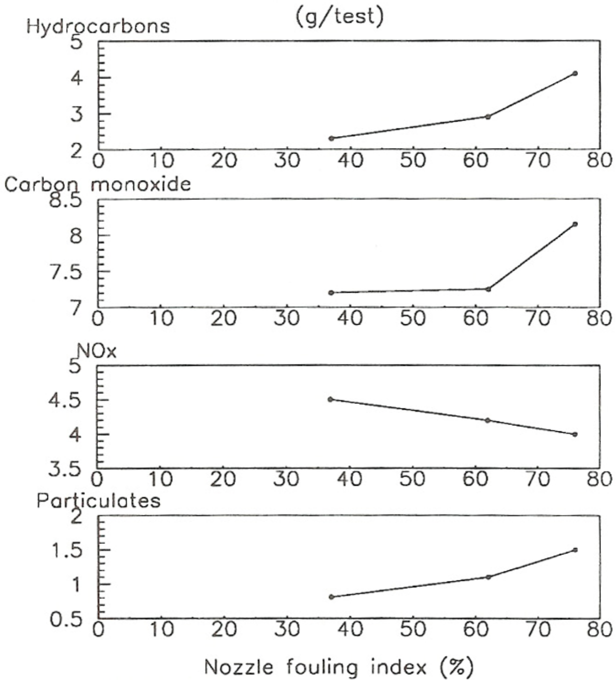

Figure 5. Exhaust emissions against nozzle fouling index taken from [24].

These results differ slightly from those presented by Sutton et al [25] where the pintle tip of the injector needle was deliberately coated, using a chrome plating technique, to simulate different levels of coking. The work by Sutton et al included results at lower levels of fouling. This showed that HC and PM emissions both increased at very low levels of fouling and it was concluded that the nozzles for this particular engine had been designed on the assumption that nozzle coking would quite rapidly reach a stable level of approximately 50% loss of flow. Earlier work by Sutton [26] had indeed shown that the level of nozzle fouling had stabilised after about 3000 km of vehicle operation.

Winterbone et al [27] considered the effect of nozzle fouling using light commercial vehicle type direct injection engine technology. Because of the change in injector technology when moving from the IDI to the DI engine technology the location of deposit build-up also changes. Figure 6 shows the deposit build-up on the injector needle; the upper picture shows a clean needle whilst the lower picture shows a needle with deposit present. The deposit build-up on the needle is extremely small except on the very tip which is below the seat area. Figure 7 shows the outside of the tip of the injector nozzle. The upper picture again shows a clean nozzle whilst the lower picture shows a significant amount of deposit build-up. Despite this deposit there was no measurable loss of flow through the injector.

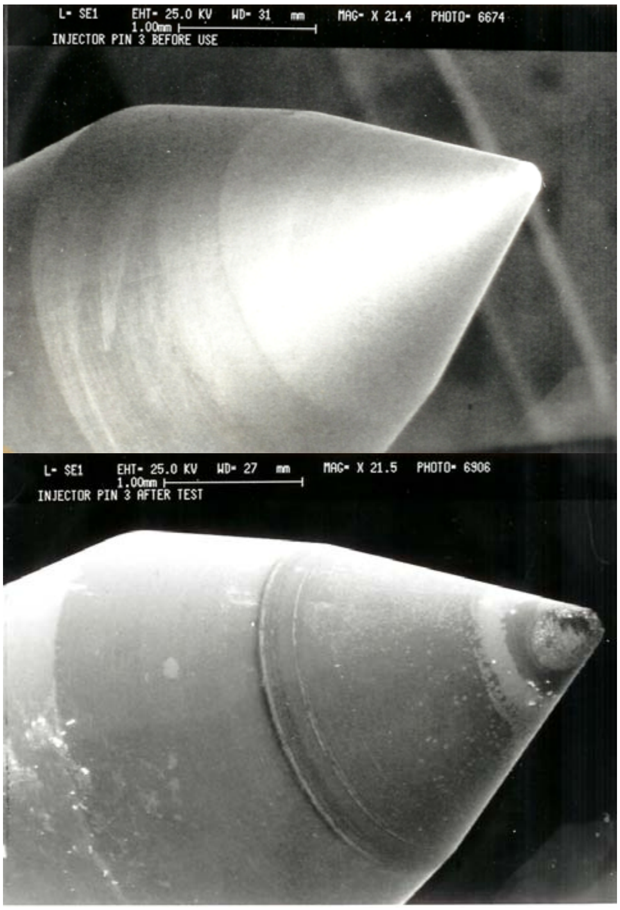

Figure 6. Clean (above) and fouled (below) injector needle [27]

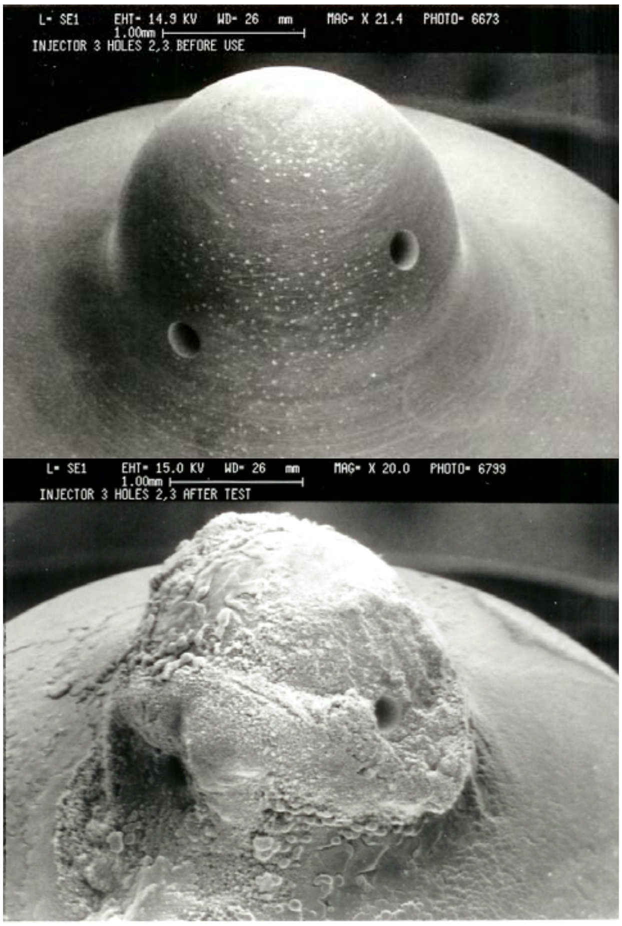

Figure 7. Clean (above) and fouled (below) injector tip [27]

A single cylinder version of a series production four cylinder engine was used for this work. It was naturally aspirated with a bore of 98.43 mm and a stroke of 127 mm. It used a Simms Minimec in-line fuel pump and CAV 6801027 FBD injector nozzles. The engine was run on a mixed duty cycle consisting of idle, medium load and high load. Figure 8 illustrates how the smoke opacity and the fuel consumption varied with time.

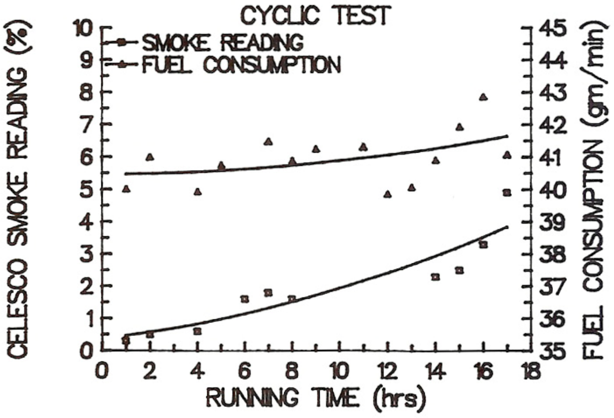

Figure 8. Smoke and fuel consumption [27]

Because there had been no measurable loss of air flow, it was assumed that performance deterioration was due to disruption of the fuel spray characteristics. Further work reported in this paper used a nozzle that had been fouled whilst fitted to an in-service diesel vehicle to investigate the effects on spray formation when fitted to a single cylinder research engine with optical access. Analysis of films taken in the optical engine showed relatively small differences in the visible spray boundary and spray tip penetration. The fouled injector did give slightly greater penetration. However the cyclic variability with the fouled injector had increased from 4% to 12% and slightly more of the fuel was being burned in the premixed regime when using the clean nozzle.

A similar piece of work reported by Richards et al [28] used a four cylinder Perkins MDi “Prima” engine to accumulate deposits and a single cylinder research engine with optical access to evaluate the effect of the deposits on the spray formation. The Perkins engine was an in-line, 4 cylinder, displacing 1994 cc with EGR for NO_X control. It was fitted with a Garret AiResearch T2 turbocharger giving 96 kPa boost pressure. The fuel injection system included a Bosch EPVE fuel injection pump and Bosch 5-hole, 2 stage fuel injection nozzles. These two stage fuel injectors were designed to give a pilot injection and a main injection. The pilot injection was produced by a 21.5 MPa initial nozzle opening pressure with a maximum lift of 0.03 mm; the main injection occurred when the injection pressure reached 35.0 MPa and there was a maximum needle lift of 0.4 mm.

In this work there was again no measurable loss of flow through the fouled injector nozzle but measurement of the period of injection for a given load was shown to increase. However, it is not clear whether this increase was due to a reduction in fuel flow rate through the injector holes or whether it was due to a change in combustion efficiency and hence fuel requirement to maintain the load set point. It was noted that although the start of heat release was constant throughout the testing the crank angle for a given percentage of heat release become later as the deposit built up on the injector. The time for 50% heat release increased from 18.9°CA to 20.5°CA.

The in-cylinder photographic work undertaken during this work showed that the deposit around the fuel injector holes was in fact having a significant effect on the spray formation. Figure 9 shows the early development of the five spray cones from a cleaned injector. Each of the four contours are at ½°CA intervals. There is one dominant spray which is a characteristic of the geometry of this particular type of injector.

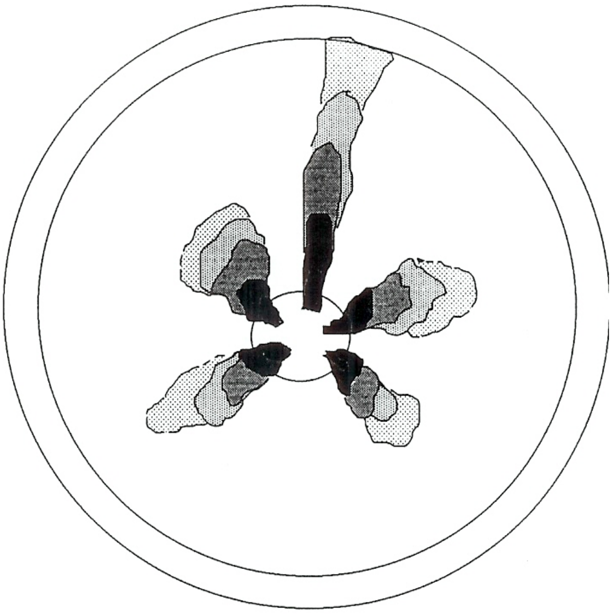

Figure 9. Clean injector spray pattern, from [28]

A corresponding image for the fouled injector is shown in Figure 10.

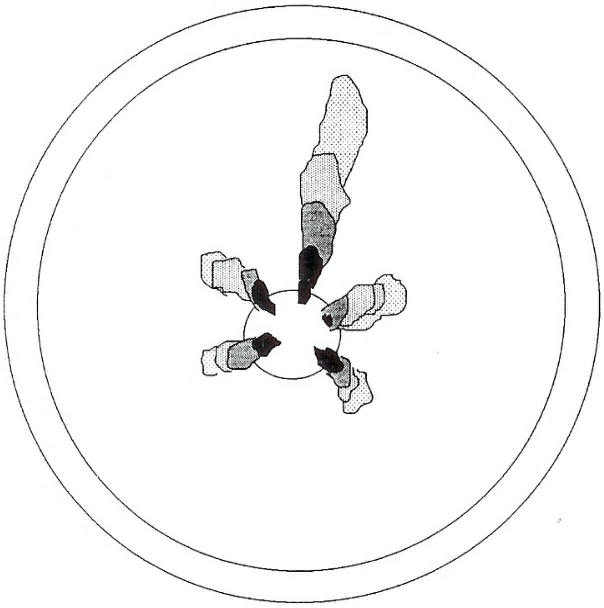

Figure 10. Fouled injector spray pattern, from [28]

From Figure 10 it can be seen that the deposit build up on the injector tip reduces the spray cone angle and the penetration. This implies a reduced flow rate through the injector hole and reduced atomisation and mixing.

Returning to the issue of running diesel engines on bio derived fuels as mentioned in the introduction; Sem conducted work to compare the injector tip deposit forming propensity of bio-ester fuels [29]. For this work he used a Yanmar 2.1 litre direct injection engine, fitted with fuel injector nozzles having four 0.23 mm diameter holes.

Figure 11 shows an injector nozzle after it had been run for 1000 hours on petroleum diesel fuel (left hand image) in comparison to the clean injector nozzle. The fouling as a result of using the petroleum diesel fuel comprised of dry flake black deposits that were not considered to be interfering with the spray formation.

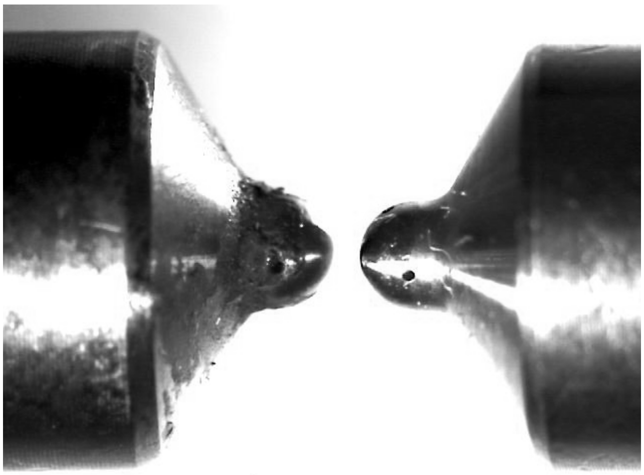

Figure 11. 1000 hrs running on petroleum diesel [29]

In contrast, Figure 12 shows the degree of fouling as a result of running the engine for 1000 hours on Soybean Methyl Ester. The resultant hard black material formed was found to be 1.4 mm at the thickest section.

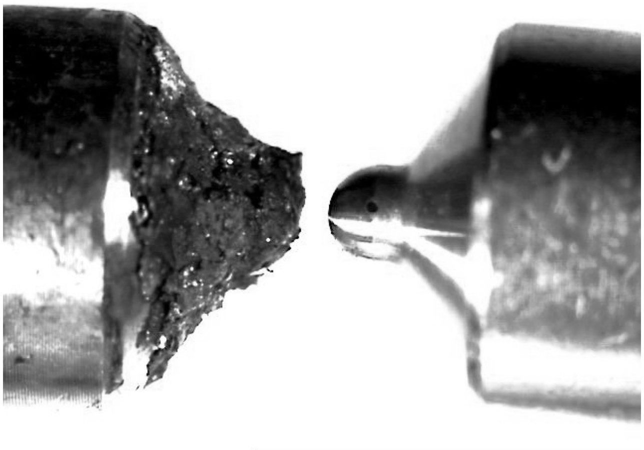

Figure 12. 1000 hrs running on soy methyl ester [29]

From SEM analysis of the material it was concluded that the deposits were most likely formed as a result of the thermal decomposition of the methyl ester molecules or the thermal decomposition of glycerine molecules that were present as impurities in the methyl ester. Unfortunately no data was presented on the effect these deposits had on the emissions performance of these engines.

Li et al [30] set out to investigate the effect of a multifunctional additive package on fuel injector deposits. Even short periods of running on vegetable oil produced a change in the level of emissions of regulated pollutants that were attributable to the formation of deposits on the tip of the injector. Li et al [30] used a Perkins Phaser 180Ti engine. This was an in-line, 6 cylinder engine of about 6 ltr displacement. It was turbocharger and inter-cooled but had no EGR. The injection system consisted of a pump-line-nozzle arrangement and the fuel injectors had six holes of 0.2 mm diameter.

Testing using the un-additised vegetable oil produced significant deposits on the injector tip within a relatively short period of operation. Figure 13 shows an SEM image of the tip of the injector. The deposit thickness around the nozzle holes was approximately 400 µm. A close up SEM image of one of the holes is shown in Figure 14.

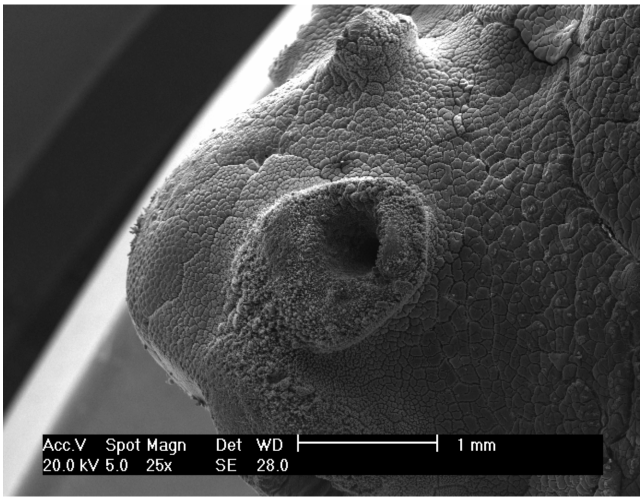

Figure 13. Injector tip having run on vegetable oil

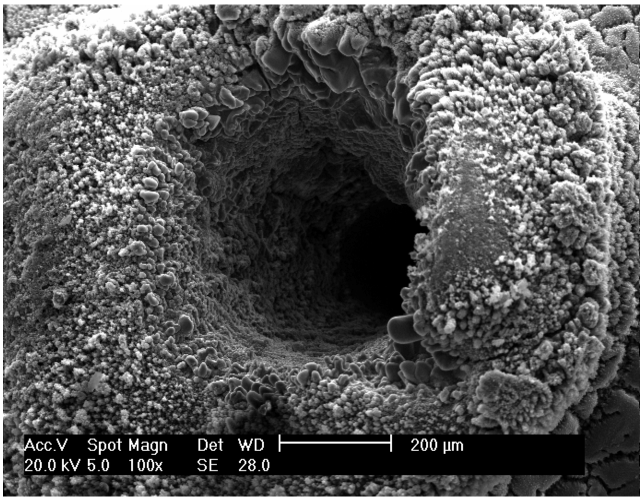

Figure 14. Injector hole deposit after running on pure vegetable oil. [30]

Emissions measurements had been taken at various points during this running and showed some significant changes. Figure 15 shows the change in PM emissions during the testing. There is a change in the PM emissions of greater than 50 % over an operating period of less than 20 hours.

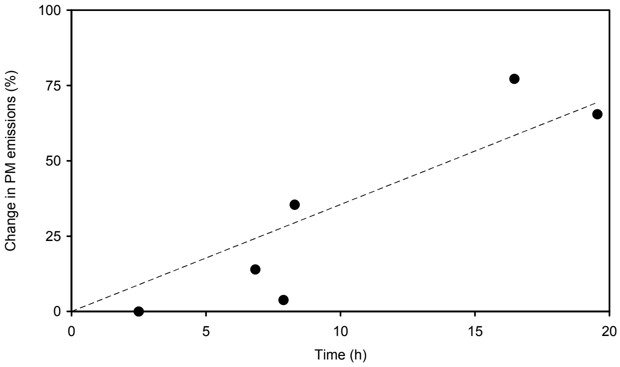

Figure 15. Increasing PM emissions with time using data from [30]

When Rudolf Diesel exhibited his engine at the Paris Expo in 1900 PM and smoke emissions were a thing of the distant future but in the current age of stringent emissions regulation it is clearly not acceptable to try and develop an engine allowing for the fact that the PM emissions could rise by over 50% within a day's operation.

EVALUATION TEST METHOD DEVELOPMENT

Whilst the need to control the smoke emissions from diesel engines had been realised at an early stage and it had been acknowledged that fuel additives had a role to play in this endeavour; the initial focus was on smoke suppression per-se [31]. However, such catalytic additives did have the secondary benefit of reducing the build up of carbonaceous deposits on the fuel injectors leading to a significant reduction in the number of engine failures due to fouled injectors [32]. By the early 1980s, fuel additives were being developed specifically to control and reduce injector deposit levels [33]. The performance of such additives and fuels in general were assessed simply from accumulated mileage under normal service conditions. There was thus a need for a short term test method that could be conducted under controlled laboratory conditions that would allow the assessment of different fuels and fuel additive combinations for their deposit forming tendencies.

It was around this time that individual companies started to develop their own laboratory test procedures to replicate the field problems in a relatively short time. This of course necessitated a recognised means of assessing the degree of fouling. Reynolds [34] proposed a method of assessing the degree of coking by measuring the loss of flow through pintle injectors. This effectively became the standard method for measuring fouling of such injectors and was used in some of the work previously discussed regarding the emissions effects of nozzle fouling.

Work by Montagne et al [23] has already been mentioned in the section regarding the effect of deposit formation on emissions performance and details of the engine technology can be found there and in the reference.

In this IFP work a short screening procedure was proposed. This procedure consisted of warming up the engine at 2000 rpm and 20% load for 20 minutes on slave injectors. These injectors were then removed and the test injectors installed. Combustion chamber pressure and needle lift data were then acquired at 1200 rpm 10% load. The engine was then run for 2 hours at the deposit accumulating condition of 1500 rpm and 40% load. At the end of this period a further set of measurements were taken at 1200 rpm, 10% load. The speed load profile for this procedure is shown graphically in Figure 16.

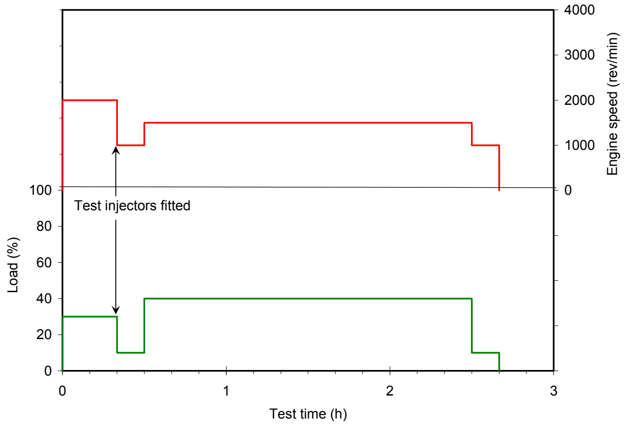

Figure 16. Screening test procedure [23]

A longer test was also proposed illustrated in Figure 17 which was considered to simulate freeway driving. This mirrored the short screening test except that the deposit accumulation mode was changed to an engine speed of 3000 rpm at 75% load and the duration was increased from 2 hours to six hours.

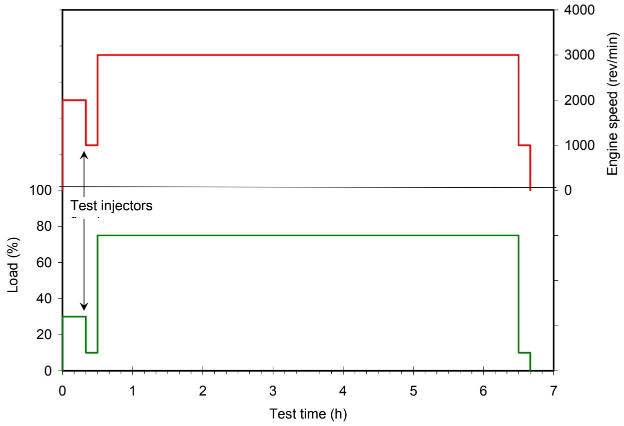

Figure 17. Longer test procedure from [23]

This test procedure became the basis for the Coordinating European Council (CEC) test procedure for IDI diesel engine nozzle coking assessment [35] often referred to at that time as the CEC PF26 procedure.

Mulard and China [35] proposed using a small generator set rather than a full engine test bench. They proposed a Kubota Z600-B engine which had only two cylinders but with a bore and stroke of 72 mm and 70 mm respectively. Each cylinder was slightly smaller than that of the XUD-9 engine used by Montagne in the CEC procedure. Further, the cylinder head of the generator was modified to minimise excessive coking at the top of the injector nozzle and to accommodate the Lucas RDNOSDC 6850 injectors as used in the CEC procedure. The generator was governed to an engine speed of 3000 rpm (50 Hz) and was loaded with two 3 kW electrical loads. As with the CEC test procedure the nozzle coking phase was of six hours duration. This test method was shown to produce very good correlation with the CEC test procedure as shown in Figure 18 which is constructed from data presented in [36].

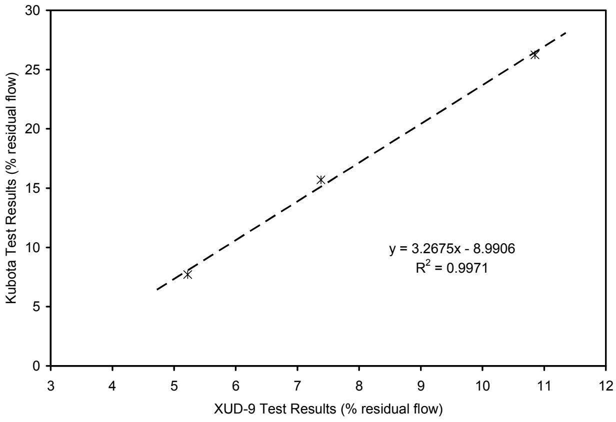

Figure 18. Correlation of Kubota and XUD-9 data, taken from [36]

Despite the low costs of this method and the good agreement with the CEC method, using the XUD-9 engine, the industry had made sufficient commitment to the four cylinder engine method to discourage change to the two cylinder engine. The six hour constant speed and load, XUD-9 test therefore became the industry standard for testing IDI injector coking propensity. However, this work did highlight some important considerations for nozzle coking tests; namely inlet air and coolant temperatures have a significant influence on coking, the shape of the fuel line pressure diagram and the initial flow of the injector can also have a bearing on the results.

The efficacy of the CEC test procedure was ensured by running round robins using a known coking and a known low coking fuel. Unfortunately, with the variation in successive batches of reference fuels and the ability of this test method to provide the necessary discrimination the implication was that this method could not be relied on and the CEC group had to develop a replacement test. The development of the revised test is detailed by Panesar et al [37].

The replacement procedure used the later version of the XUD-9 engine designated as the XUD-9 A/L and the six hour constant speed load test was replaced with a ten hour cyclic test, details of which are shown schematically in Figure 20.

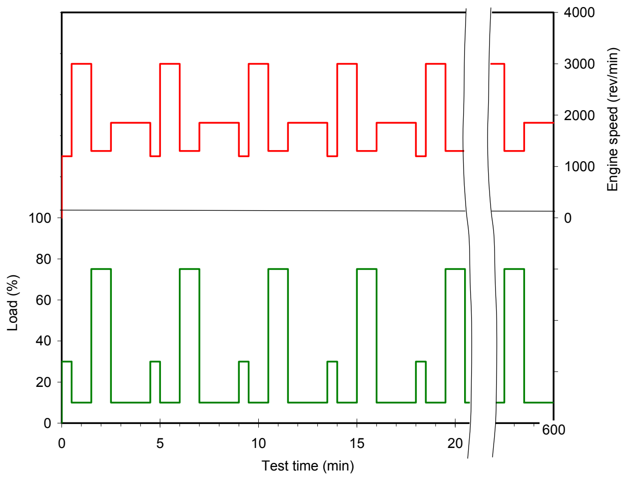

Figure 20. 10 hour XUD-9 A/L test cycle. [37]

The other major difference in the new procedure was the injection timing. The automatic timing adjustment mechanism within the fuel injection pump is locked and the dynamic fuel injection timing is adjusted according to a well defined set of criteria. The dynamic timing is then set within these criteria to produce a specified level of nozzle coking for a calibration fuel. Thus, as successive batches of the calibration fuel are produced then changes in the severity of these fuels can be accommodated by variations in the dynamic timing. This ability to adjust the engine to match changes in fuels has allowed this test method to continue as an industry standard for more than ten years now.

Despite the robustness of the XUD-9 A/L test for assessing the nozzle coking propensity of fuels in an IDI diesel engine, it was felt that with the spread of direct injection (DI) technology to the passenger car fleet a test method was required that addressed the nozzle coking propensity of fuel in this newer engine technology.

As noted in the previous section; earlier work on DI injector fouling had shown that loss of flow through the injector nozzle was not as important or reliable a metric as it had been for IDI engines. Despite the concerns of the motor manufacturers and reported field problems it proved extremely difficult to reproduce these problems under controlled laboratory conditions. As noted by Hawthorne et al [38] “Previously, the industry attempted to develop a direct injection engine test for fuel evaluation centered on the Ford Duratorq engine. However the proposed test was rejected primarily due to the required addition of zinc neodecanoate to the fuel to increase test severity and induce injector fouling.”

Birgel et al [39] used a single cylinder research engine and confirmed that zinc doped fuel is significantly more prone to deposit formation than non-doped fuel. Arpala et al [40] used a four cylinder engine and also confirmed the adverse effects of zinc. One of the justifications put forward for the inclusion of zinc neodecanoate is that although zinc is not present in fuel leaving the refinery it does get picked up in the fuel supply chain and in the vehicle. However, Caprotti et al [41] showed that the level of zinc in fuel samples taken from a vehicle over a 50,000 km field trial was always below the detection level.

Even though there are still concerns about the inclusion of zinc compounds as dopants, the CEC group have developed a test procedure using a Peugeot DW10 ATED engine that uses fuel doped with zinc neodecanoate. The engine is run to a cyclic test regime that is described in [38] and shown graphically in Figure 21 for completeness.

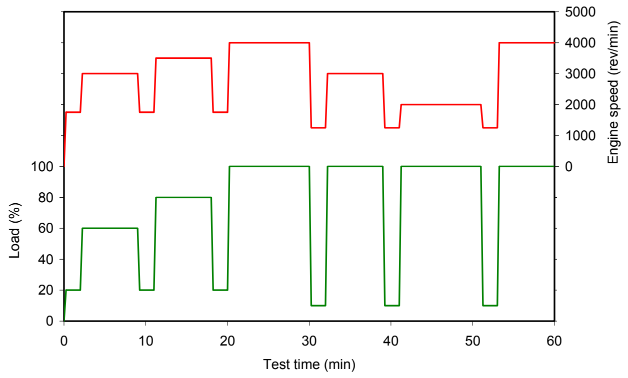

Figure 21. DW10 test cycle. [38]

The one hour cycle shown in Figure 21 is repeated eight times. The engine is then shut down and allowed to soak for eight hours and then restarted and the cyclic operation instigated for a further eight hours. These latter two steps are then repeated to give a total engine run time of 48 hours interspaced with 40 hours of engine shutdown. Hawthorne et al [38] concluded that this was “equivalent to what a vehicle would experience over a full life time.” This is currently the only industry standard test to assess the deposit forming potential of fuels in a DI diesel engine.

The preceding discussion centred on engines used in light duty vehicles and passenger cars which had predominantly been of the IDI design. It had been noted that “To evaluate detergent additives, in most of the reported dynamometer tests, prechamber type IDI engines have been used. Although most of the diesel fuel is used in heavy duty DI engines, no DI engine dynamometer test has been reported in the literature for rating fuel and additive injector deposit characteristics.” Virk et al [42] had tried to rectify this situation by proposing a test method using a single cylinder research engine with a cylinder capacity of 688 cm^3 and compared this with the performance of a vehicle with a four cylinder engine with a cylinder capacity of 981 cm^3. Whilst these engines were of the DI design their cylinder sizes were at the lower end of the range that could be classified as heavy duty.

Gallant et al proposed a method using a Cummins L10 engine [43]. The Cummins L10 engine had begun development in 1976 [44] and utilised the Cummins PT® fuel system. The cylinder capacity of the L10 engine was 1667 cm3 giving a total engine capacity of 10 litres. This fuel system contained a fuel injection nozzle which was claimed to have a “novel means for substantially preventing products of combustion from entering the injector and passing to critical parts thereof or to fuel lines connected to the injector." [45] The tip of the injector is shown in Figure 22, taken from [45]. In this figure the plunger is held in the charging position by the force of the return spring (not shown in the figure). Fuel enters the tip of the injector, through the passage labelled 98, to fill the chamber labelled 101.

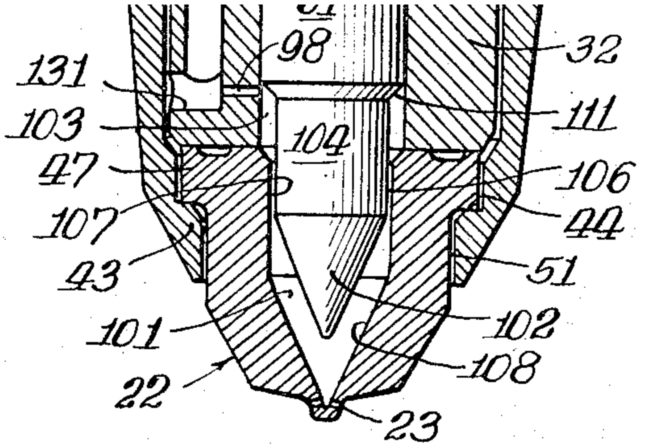

Figure 22. L10 injector, charging position [45]

During the injection event, the plunger is driven, against the return spring, by a cam and push-rod mechanism to the position shown in Figure 23, again taken from [45]. This forced the fuel from the chamber at the end of the nozzle into the combustion chamber through the nozzle holes labelled as 23.

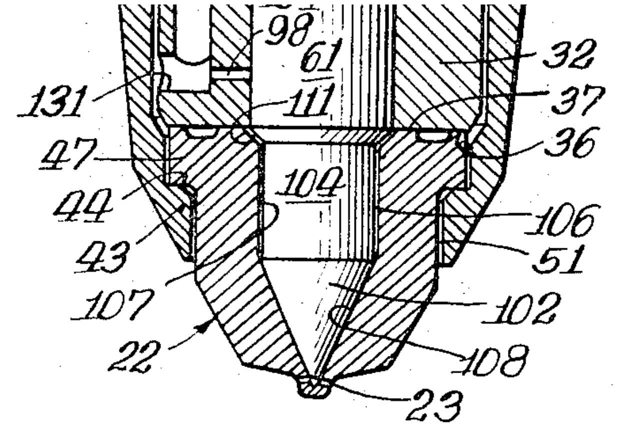

Figure 23. L10 injector, injection position [45]

After the injection event, the plunger returns under the action of the return spring, to allow the chamber to refill with fuel. In all probability, during this return stroke some combustion chamber products were drawn into this volume allowing them to mix with and possibly react with the incoming fuel for the next injection event. In the procedure developed by Gallant et al [43] this was the area of the plunger on which deposits were formed. The test procedure consisted of connecting two L10 engines together, nose to tail, without a dynamometer. One engine was run to motor the other engine and every 15 seconds the engines switched from motoring to driving. Engine speed was controlled by adjusting the high idle governor on the fuel pumps and the engines were then run at either full rack or closed rack. The load and speed cycle is thus as shown in Figure 24. This cycle is repeated to give a total test duration of 125 hours.

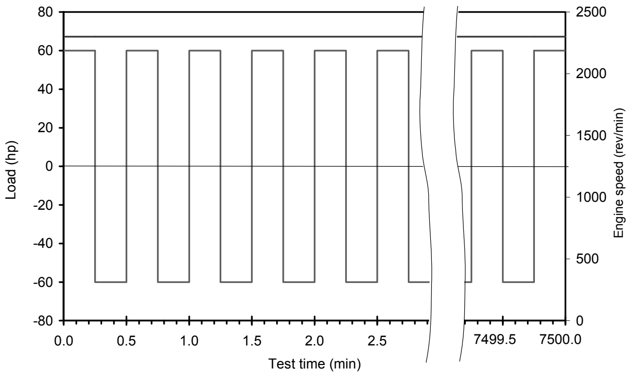

Figure 24. Cummins L10 test cycle. [43]

The degree of fouling is determined by flow loss and visual rating of the deposits on the injector plunger. Blythe and Flask [46] proposed an image analysis system for rating the injector plungers but this never became part of the recognised method.

Although the Cummins L10 test procedure became the *de facto* standard for DI injector deposit formation there were other proposals for a less expensive and quicker test procedure that could be used for screening purposes. One example was that proposed by Gutman et al [47] which used a Petter AD-1 engine. This, like the engines proposed by Virk et al was naturally aspirated but had an even smaller cylinder capacity than that proposed by Virk et al. The cylinder capacity of the AD-1 engine was only 367cm^3. The proposed operating cycle did have similarities to the L10 procedure in that it was highly cyclical. Following a 10 minute idle period the engine was alternated between a low load (about 45%) and a high load (about 75%); for 10 minutes at each load. After five hours of operation the engine was stopped and allowed to soak for an hour. This was then repeated to give a total test duration of 23 hours; four periods of running and three soak periods. This test cycle is shown schematically in Figure 25.

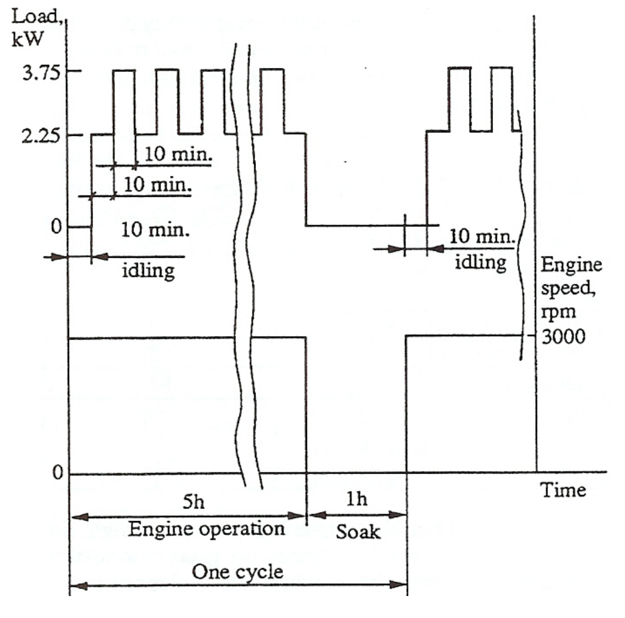

Figure 25. Proposed Petter AD-1 test cycle. [47]

Because the Cummins L10 engine design was about fifteen years old when Gallant et al developed the procedure it was inevitable that the engine would eventually become obsolete. This is now the case and since then there has been no recognised procedure for heavy duty diesel engine nozzle fouling. A few years ago, Williams [48] proposed a procedure using an engine with an individual cylinder capacity of 933 cm^3. The procedure was again highly cyclical and designed to represent a hill climbing drive cycle; like the L10 procedure this included over-run conditions. The speed and load conditions of this cycle are shown schematically in Figure 26. The total test duration of 20 hours shown in Figure 26 was proposed for a pre-Euro I engine whilst a 40 hour test was proposed for a Euro I engine.

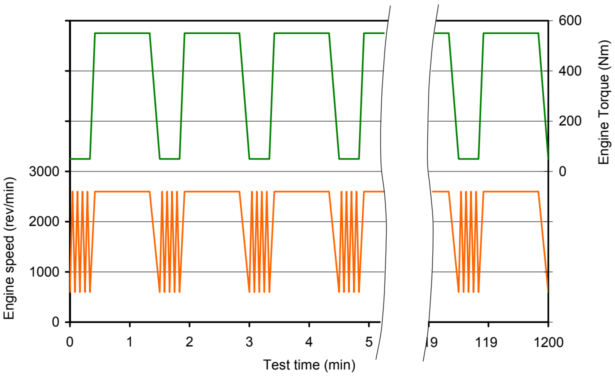

Figure 26. Heavy duty engine test cycle. [48]

The difference in test duration was proposed due to the difference in injection nozzles used in the two different engine specifications. The pre-Euro I engine had a 4-hole injector with a larger sac volume and hole diameter than the 5-hole injector used in the Euro I engine. Williams concluded "The initial similarity established between test generated and road generated needle deposits, coupled with the apparent test response to detergent is evidence that the test is appropriate for use in rating diesel fuels and detergent additives used to combat injector fouling." However, this procedure has not gained wide support. This may be influenced by the fact that the Mercedes OM366LA engine technology proposed by Williams is now more than twenty years old and is not representative of the high pressure common rail engines now being produced.

Therefore, there is no widely accepted method for assessing deposit formation in current technology heavy duty diesel engines. There is thus a pressing need for the industry to develop a test procedure that adequately assesses the deposit forming propensity of fuels in modern heavy duty diesel engines.

CURRENT ISSUES

The formation of deposits within the fuel systems of US diesel engined equipment continues to cause the industry serious concerns. Problems do not appear to be restricted to any particular engine type or geographic region, making it extremely difficult to isolate cause and effect. As noted above there is no industry standard for assessing the propensity of a fuel to form deposits within a heavy duty diesel engine since the demise of the L10 test procedure. For light duty diesel engines the only industry standard procedure relies on a reference fuel that is artificially doped with a zinc compound. This recent bout of reports of injector fouling has been accompanied by an increase in reports of fuel filter fouling. This suggests that deposits or deposit precursors are forming in the fuel or that by some means deposits formed in the injector are being re-circulated to the fuel tank.

PREVIOUS WORK

It has been suggested [49] that "Historically deposits have been generated from a number of sources: bio-contamination, both aerobic and non-aerobic, water contamination, lube oil adulteration, additives, dirt, metals in fuel, and biodiesel degradation. These may be ascribed to "poor housekeeping," incorrect additivation, deliberate adulteration or some combination." Cook and Richards [50] surveyed some of the possible causes of deposit formation in fuel systems brought about by the oxidation of the fuel components leading to the formation of deposit precursors. Work by Ullmann et al [51] suggested that interactions may occur between mono acid lubricity improver and detergent additives. However, to produce the proposed reactions the authors used a large excess of one of the reactants involved in the production of the detergent. This is in effect poor housekeeping on behalf of the additive producer. Also Schwab et all [52] stated "- in some applications where monoacid lubricity improvers were used, there were no performance issues related to injector deposit formation." Schwab et al also claimed "Tests on the thermal stability of problematic fuels showed them to be very stable." In response to this they proposed that one possible cause of problems was sodium salts of alkenyl succinic acids that were insoluble in ULSD. Such deposits were reproduced in the laboratory but again required atypical levels of sodium to be present. This is again, an indication of poor housekeeping.

Work in collaboration with the University of Nottingham, used the Hydropyrolysis technique [53] to showed that fuel filter deposits were a complex mixture of graphitic carbon, polyaromatics, cycloalkanes, aromatics, straight chain and substituted alkanes, and acids. The occurrence of sodium salts was limited. Later work with the University of Southampton [54] used a combination of Gas Chromatography with Mass Spectrometry detection (GC/MS), Fourier Transform Infra-red analysis (FTIR), Inductively Coupled Plasma spectroscopy (ICP), Nuclear Magnetic Resonance spectroscopy (NMR) and elemental analysis to characterise injector deposits, and fuels.

The combination of high resolution mass spectrometry, and ICP was used to analyse fuels. Results of metal analysis by ICP of two fuels with no history of deposit formation and three fuels that are known to cause deposits is given in Table 1.

None of the fuels tested showed any species indicative of the presence of biodiesel.

It can be seen from Table 1 that there is no correlation between the presence of calcium and sodium and a propensity to form deposits. It also clear that the presence of zinc is not common nor is it necessary for the promotion of deposit formation. Table 1 also shows that neither fuels 3 or 4 contain any significant amount of any metal yet fuel 3 has a propensity to form deposits whereas fuel 4 does not.

Table 1 Metal analysis by ICP.

Fuel	1	2	3	4	5
Ca (mg/kg)	2.5	**1.7**	**<0.1**	<0.1	**1.8**
Fe (mg/kg)	0.3	**0.1**	**<0.1**	<0.1	**0.2**
Mg (mg/kg)	0.3	**0.3**	**<0.1**	<0.1	**0.3**
Si (mg/kg)	0.5	**0.3**	**<0.1**	<0.1	**0.1**
Na (mg/kg)	1	**0.7**	**<0.1**	<0.1	**1**
Zn (mg/kg)	<0.1	**<0.1**	**<0.1**	<0.1	**0.2**
Coking	No	**Yes**	**Yes**	No	**Yes**

The previously mentioned spectroscopy techniques have been used to identify differences between deposit forming and no-deposit forming fuels. Fuels 1 and 5 from Table 1 were chosen as two fuels with similar levels of metal contamination but opposite trends in terms of deposit forming tendency.

Various mass spectrometric techniques were used to look for compositional differences between these two fuels. Low resolution electrospray mass spectrometry (LR-ESI-MS) analysis of Fuel 1 showed an abundance of ions at m/z 317 in the non-deposit forming fuel. The most likely assignment of molecular formula was thought to be $C_{19}H_{34}O_2Na$. This was confirmed by the accurate mass report (Figure 27) produced by Fourier Transform Ion Cyclotron Mass spectrometry (FTICRMS).

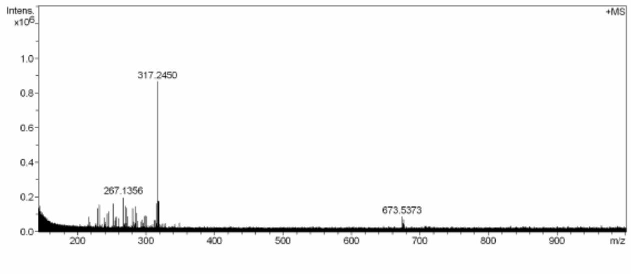

Figure 27. Accurate mass report for ion at m/z 317 in Fuel 1

This hypothesis was also consistent with the presence of species of relative molecular mass 294 in the GC-MS data for this fuel. A reconstructed ion chromatogram (RIC) was used to identify the ion at m/z 294. Similar analysis of Fuel 5 did not indicate the presence of the C_{19} acid species. Analysis of both fuels found that they contained a high molecular weight material with ions at m/z 673. This observation along with the lack of ions associated with carboxylate sodium salts of dimer, dodecylsuccininc and hexadecenyl succinic acids demonstrates that it is not necessary for the presence of sodium carboxylates in a fuel for deposit formation to occur. The high molecular weight material at m/z 673 may be sodium stearyl palmityl tartrate which is used as an emulsifier.

Work continues to understand these issues around the common rail, internal injector deposits and the precursor chemistry as well as additive solutions and these will be the subject of future publications.

LATEST WORK

To build on the understanding from previous work, a fuel injector was obtained from the field, having been removed from the engine after the operator had reported operational problems. Three fuel filters were also obtained from the field on separate occasions, again after operators had reported operational problems. Both the fuel filters and the fuel injector were from heavy-duty diesel engines in operational service in North America. The problems manifest as power loss resulting from needle sticking, injector failure, and filter blockage. All of the vehicles had been operated on current specification ULSD fuel.

In an attempt to understand the nature of these deposits, application of a novel hydropyrolysis technique (previously applied to filter deposits [53]) was used to investigate the deposits from the injector tip and the latest three fuel filters. The filter deposits were isolated as described in [49] and the injector tip deposits were scraped from the tip and subjected to the same protocol.

The hydropyrolysis technique is described in [53] but the salient points are repeated here for conveniance The collected solid was mixed with the sulphided molybdenum catalyst prior to hydropyrolysis. The reactor was first heated from ambient temperature to 250°C at a rate of 300°C/min and then to 350°C at a rate of 8°C/min. A pressure of 150 bar of hydrogen was maintained in the reactor and a gas flow of 5 l/min swept any products into a silica trap that was cooled by dry-ice. The rig was allowed to cool and the silica was removed from the trap for subsequent analysis. Fresh silica was placed in the trap and the sample was subjected to hydropyrolysis under the same pressure and hydrogen flow rate as before. The sample was heated from ambient to 350°C at a rate of 300°C/min; any material that would be driven off below this temperature would already have been driven off and collected in the previous batch of silica. The sample was then heated to a final temperature of 520°C at a rate of 8°C/min. This higher temperature was held for 2 min. After allowing the rig to cool this second batch of silica was removed. The products collected in each batch of silica were desorbed from the silica using dichloromethane (10ml) and then analysed by GC-MS.

The GC-MS analysis was performed on a Varian CP-3800 gas chromatograph operating in full scan mode, interfaced to a 1200 mass spectrometer (EI mode, 70 eV). Separation was achieved on a VF-1MS fused silica capillary column (50 m x 0.25 mm i.d., 0.25 μm thickness), using helium as the carrier gas, and an oven programme of 50°C (hold for 2 min) to 300°C (hold for 20.5 min) at a rate of 4°C/min.

RESULTS

Analysis of the fuel filters

Figures 28 to 30 show the total ion chromatogram (TIC) of the hydropyrolysis products from filters 1, 2, and 3 respectively. The upper trace in each figure shows the TIC for the material desorbed up to a temperature of 350°C and the lower trace shows the TIC the product trapped by the silica during the hydropyrolysis from 350°C to 520°C.

In all cases, the quantities of material physically trapped by heating the sample to 350°C were significant and the hydrocarbon compositions were generally similar to those obtained at the higher temperature of 520°C.

It can be seen from Figure 28 that the TICs for the products from filter 1 are dominated by *n*-hexadecane and *n*-octadecane with smaller contributions from other C_{12} to C_{22} *n*-alkanes. From experience, it is likely that a fatty acid accounts for the elevated proportions of n-C_{16} and n-C_{18} observed in both the low and high temperature hydropyrolysis products. The carboxylic acid group begins to decompose at a low temperature and hence begins to yield corresponding *n*-alkanes below 350°C.

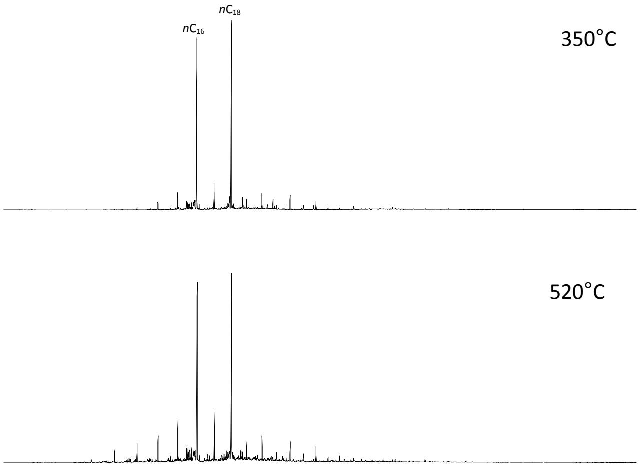

Figure 28. TIC of residue from filter 1

The products from the hydropyrolysis of material from filter 2 contain occluded relatively heavy hydrocarbons ranging from C_{18} to C_{26} *n*-alkanes. Bound aromatics are released during the hydropyrolysis up to 520°C and are thus present in the TIC in the lower trace of Figure 29. The latter are indicative of a lightly cross-liked macromolecular phase but the similarity in the C_{18} to C_{26} *n*-alkane distributions indicates that those present in the higher temperature product could represent the remnants of the physically entrapped hydrocarbons present in the particulates. Clearly, the hydrocarbons have a significantly higher boiling range than the diesel fuel thus indicating the contribution from a heavier fraction.

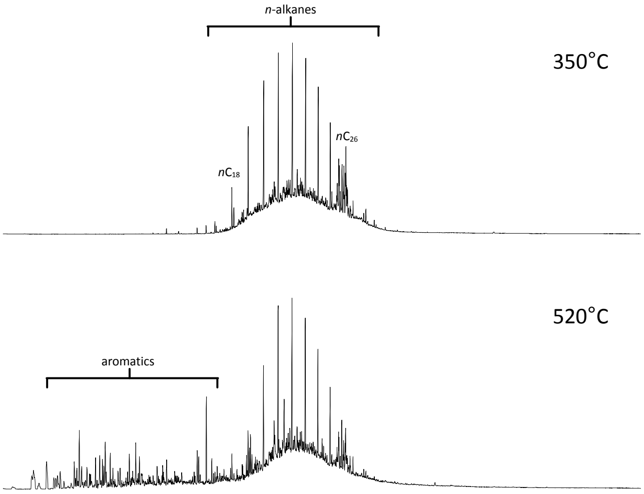

Figure 29. TIC of residue from filter 2

As shown in Figure 30, the TICs for the products from filter 3 indicate that the hydropyrolysis products from these deposits are dominated by a series of steranes and sterenes.

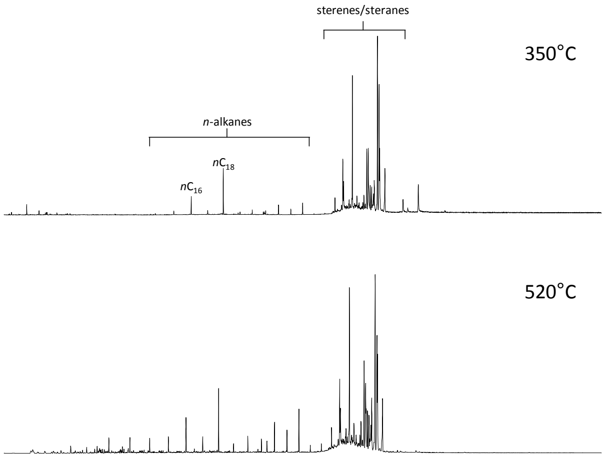

Figure 30. TIC of residue from filter 3

The steranes and sterenes are most likely derived from steryl glycosides by cleaving the relatively weak C-O bonds at relatively low temperatures. Steryl glocosides are often found as impurities in biodiesel fuel [55]. These compounds have previously been identified [55, 56] as a potential source of “above the cloud point” precipitate leading to filter blocking. An example of the structure of a steryl glocoside is shown in Figure 31.

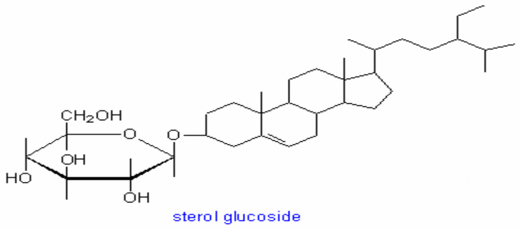

Figure 31. Structure of a steryl glucoside

In addition, *n*-alkanes with a distinct even/odd domination (especially nC_{16}/nC_{18}) are evident. As for previous samples [53] these are fatty acid in origin. A heavy fraction of diesel accounting for C_{18} to C_{26} is found in the case of filter 2. Not only is there the presence of carbonaceous deposits and acids as described in a previous publication [53] but also residual steryl glucosides which have previously been implicated in diesel filter fouling [55]. Thus, there are several possible fouling mechanisms responsible for the deposits on the three filters and they are present at the same time.

Analysis of the fuel injector tip

Figure 32 shows the TIC and SIC (m/z 71) traces for the hydropyrolysis product for the deposits removed from the injector tip. This shows a complex distribution with a C_{18} dominating peak. Again this is associated with carboxylic acids, and it links to what has been seen on filters. Note the phthalate peak is contamination from the plastic bag the injector was received in.

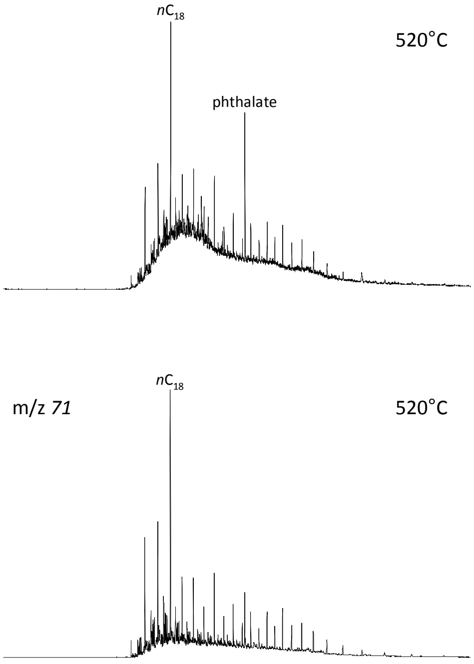

Figure 32. TIC (upper trace) and SIC (m/z 71) (lower trace) of injector tip residue

Figures 33 shows the GC-MS single ion chromatogram (SIC) data for alkylbenzenes, alkylnaphthalenes and alkylphenanthrenes for the sample from the injector tip. For comparison, Figure 34 shows the corresponding TICs from the sample derived from the fuel filter deposits presented in [53]. The distributions of these species in the fuel injector tip sample are extremely complex when compared to the earlier fuel filter sample investigated [53]. Hence, it can be concluded that the aromatics released by hydropyrolysis of the injector tip sample are of relatively small ring size and heavily alkyl substituted with no unsubstituted PAHs being observed. This is in surprising contrast to the fuel filter residues where the postulated intermediate types in the transformation from fuel to carbonaceous deposit by a hydrogen abstraction mechanism are seen [53]. This may be an indication of the rapid degradation of the fuel brought about by the high temperatures and pressures it experienced in the high pressure fuel system.

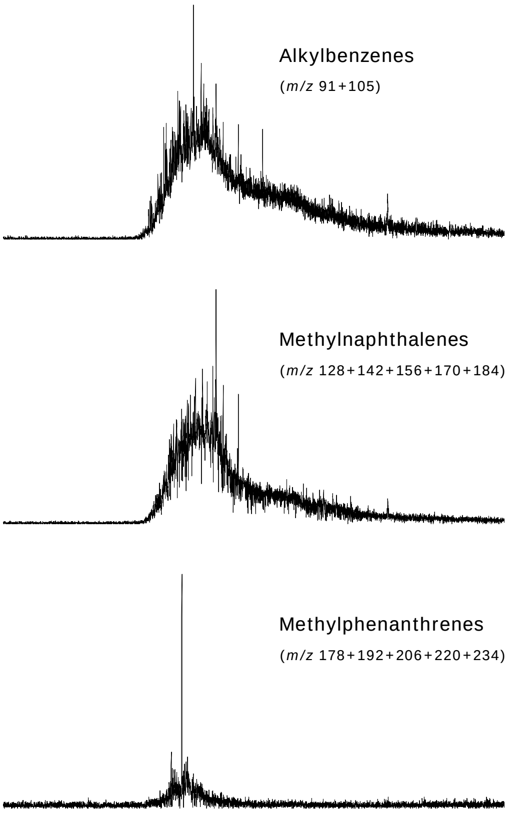

Figure 33. Characteristic SICs of injector tip residue

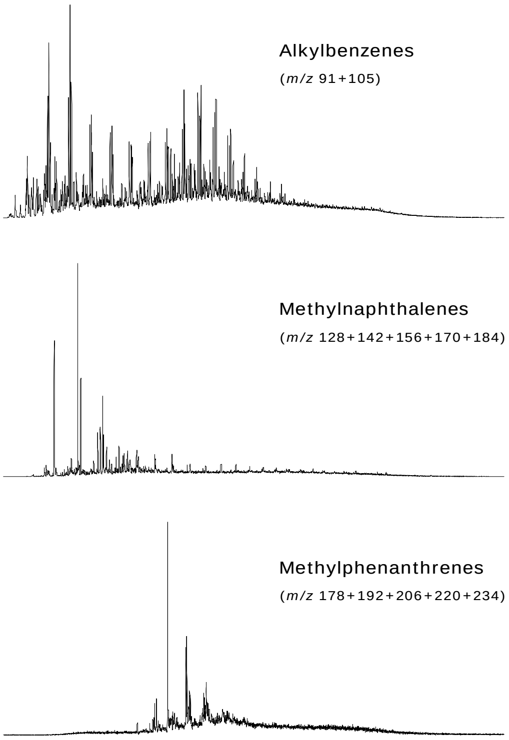

Figure 34. Characteristic SICs of fuel filter residue reference [53].

CONCLUSIONS

It is likely that deposits have been forming in the fuel delivery systems of diesel engines ever since there inception at the end of the nineteenth century. Due to the tolerance of diesel engine designs and the absence of significant emissions regulations this was of only minor concern until the latter part of the twentieth century. At this time, work was performed to identify possible causes of the deposits being formed and to develop test procedures to quantify the propensity of different fuels and fuel/additive combinations to control this deposit formation. A test method using a passenger car diesel engine was developed in Europe and a method using a heavy duty engine was developed in the US. A further test method using a passenger car diesel engine with a high pressure common rail fuel system has also been developed. By use of these test methods, fuel and fuel additives have been developed that in conjunction with good housekeeping have aleviated the majority of field problems.

There has been a recent resurgence in reported field problems and a great deal of effort is being applied to try and understand the mechanisms leading to this fresh outbreak. Some of the latest work, including that reported here, has shown that the mechanisms causing these issues are complex and undoubtedly not of a single origin. However, similarities in the species found in current deposits do show common links between both fuel filter and fuel injector deposits. Work continues to understand the mechanism of formation of these deposits and the role of fuel properties and deposit control additives in inhibiting their production.

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