

Internal Fuel Injector Deposits

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ABSTRACT

The need for improved emissions and fuel economy are placing increasingly severe performance requirements on compression ignition engines. These are satisfied in part by advanced fuel injection equipment that provide multiple injections and increased injection pressures along with higher operating temperature. Fuel composition is also changing, with increased use of non-traditional feedstocks combined with a range of additive chemistries to restore or enhance fuel quality.

Within this environment, a number of worldwide automotive companies have noted a trend towards increased Internal Injector Deposits (IID). Little quantitative information to understand the root cause is available, largely due to difficulty in reproducing the issue under controlled conditions. The present study details the results of an accelerated test methodology, which is used to evaluate the interrelated effects of fuel composition and operating environment. In general, the results indicate low deposit tendencies with pure hydrocarbons and high quality Fatty Acid Methyl Ester (FAME). The results identify a number of potential deposit precursors that are likely to be responsible for soft metal carboxylate soap deposits and also hard polymeric lacquers.

INTRODUCTION

Diesel engine technology has evolved rapidly in the past decade. Engine manufacturers have striven to optimize the combustion process to simultaneously improve fuel consumption and engine out emissions. These developments have been made possible in part by advances in Fuel Injection Equipment (FIE). One of the consequences is the need to achieve better atomization and penetration of the fuel charge, with injection pressure approaching 300 MPa in some applications; combined with

multiple pre- and post-injection strategies. Performance and emissions requirements must be maintained throughout the vehicle's life, which for heavy duty vehicles can be up to 1,000,000 miles (1.62 million km).

Higher injection pressures require close tolerances and significantly increase operating temperature. The theoretical average temperature rise during release of fuel within the injector control mechanism or fuel recirculation is proportional to rail pressure. During rapid energy changes transfer to thermal energy results in a flash temperature rise within the liquid before the energy is dissipated to the surrounding metal. An approximate calculation using equation 1 indicates that an adiabatic temperature rise of approximately 115°C will occur upon release of fuel at 200 MPa. This temperature rise would be superimposed on the bulk temperature within the injections system, which may be 50 to 90°C.

$$\Delta T = \frac{P}{\rho \times C_v} \quad (1)$$

Where

ΔT = Temperature rise, °K

P = Pressure, Pa

ρ = Fuel density, kg/m³

C_v = Specific heat capacity of fuel, J/kg/°K

Coincident with these hardware developments, the range of fuels marketed for use in compression ignition engines is developing. Increasingly severe refinery process to reduce sulphur content has reduced the ability of the fuel to carry semi-soluble reaction products, while at the same time necessitated the use of lubricity additives and in some instances corrosion inhibitors. The use of FAME is now commonplace in many regions, while some marketers have responded to the possibility of nozzle-hole coking in fuels containing FAME by increasing the concentration of Deposit Control Additive (DCA).

The above mentioned trends result in an increasingly severe operating environment, with the potential for deposit formation at a number locations as shown in Figure 1 [1, 2, 3]. The issue of nozzle-hole deposits has been addressed through use of DCA and is relatively well understood through coordinated research by the fuels, additives and hardware manufacturers. Incidence of Internal Injector Deposits (IID) is increasing and has reached a level that requires coordinated industry study [4, 5].

In many instances it is difficult to identify which fuels are responsible for creating IID and virtually impossible to obtain representative fuel samples for analysis. Similarly, analysis of IIDs on field returns are difficult to interpret with certainty, as many are insoluble in conventional solvents

and consist of a complex mixture of materials, some of which may have been inadvertent contamination during removal and disassembly. As a result, the few published studies are often mutually contradictory.

The present study details the composition of the various deposits observed in practical operation. A screening test is then used to replicate IIDs under controlled conditions, with the objective of defining potential range of fuel components, contaminants or additives that may be responsible for these issues. Not all such deposits will be observed in the field and the objective was not to replicate any single instance of field deposit. However, the results confirm the general observations from field returns described in this and other studies.

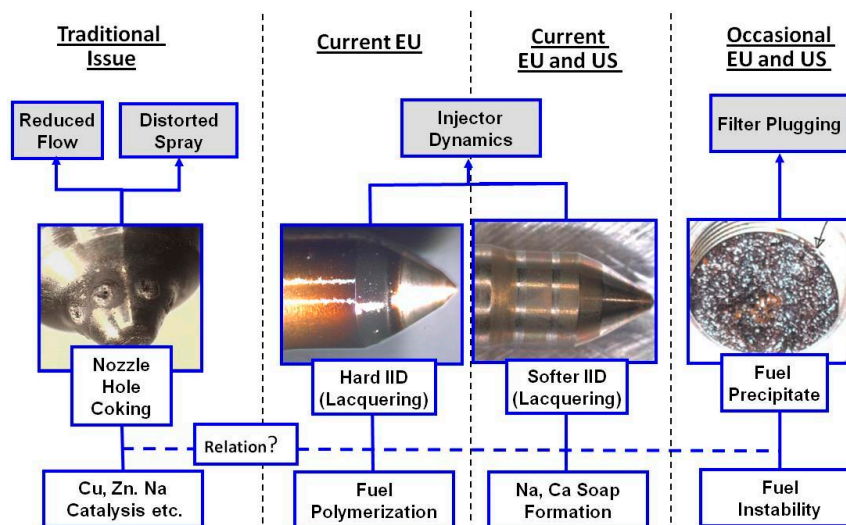


Figure 1 – Examples of Typical Fuel Derived Deposits

NOZZLE-HOLE DEPOSITS

Historically, most deposit issues occurred in the injector nozzle-holes, thereby restricting fuel flow and affecting injection quality [6, 7]. It is likely that liquid fuel stored in the injector tip is heated during the combustion process and expands during the expansion stroke. A combination of evaporation of lighter fractions and degradation leads to sticky deposits. The process is affected by elemental fuel contaminants, soot, reactive combustion products and volatilized lubricating oil

As a result, nozzle-hole deposits are difficult to model theoretically or to reproduce using non fired engine tests. Fortunately, engine tests are available, with older FIE designs reflected by the indirect injection (IDI) XUD-9A engine from PSA according to the CEC F-23-01 test procedure, while newer designs are represented by the CEC procedure for the "DW10" engine which uses a common rail injection system. Three main influences on the creation of nozzle-hole deposits are described in the literature: operating temperature, fuel composition (especially zinc) and nozzle-hole geometry [3, 8, 9, 10,

11]. It has been postulated that zinc acts as a catalyst or forms a fatty acid soap [5] although others suggest that it is simply precipitating out as zinc oxide.[10] The use of FAME appears to produce varying levels of nozzle-hole fouling, with no clear position as to why different biofuels generate different levels of deposits [3, 10].

The issue is likely to have been further aggravated by smaller nozzle-holes and high efficiency honed or tapered holes, while modern combustion and air management systems tend to increase temperature with a concomitant increase in deposits. However, in practice, the issue is being addressed using suitable deposit control additives, combined with optimized nozzle-hole design which tends to create cavitation [11]. As a result, deposit within the nozzle-holes is not addressed in this paper.

INTERNAL INJECTOR DEPOSITS (IID)

BACKGROUND - IIDs are being reported within the fuel system, typically within the injector body close to areas where fuel pressure is released either through normal fuel flow or internal leakage. Such areas also tend to be close

fitting components in relative motion and so are potentially affected by IID accumulation. Sporadic issues have been reported by all FIE manufacturers, indicating that the process is associated with occasional variation in fuel composition [5]. The effects of IID appear consistent across all injection manufacturers, typically affecting injector dynamics resulting in a measurable change in engine performance that is readily apparent to the operator [4, 5].

The mechanisms for IID formation are likely to be different to those observed in nozzle-hole coking, since there is no possibility of exposure to combustion gasses or evaporation of fuel from the wetted surface. Instead, it is likely that precipitation of microscopic insoluble materials occurs onto nearby surfaces. It is unlikely that the metallic surfaces play an active role, since the IID may be many microns thick effectively shielding the surface from the reaction process.

Two broad categories of IID have been identified, as shown in Figure 1:

- Soft opaque deposits that are often water soluble primarily comprised of metal ions, typically sodium carboxylate soaps.
- Hard often brown transparent deposits that are insoluble in most solvents (except strong bases and carboxylic acids) and appear to be formed of polymeric material.

Some studies have further subdivided the hard polymeric deposits into those produced by fuel oxidation products and additive related constituents [4]. In general, soap related deposits are most prevalent. Issues have occurred in the past with soap based deposits, generated by metal containing additives present in crankcase oil reacting with dimer acid based fuel additives [12, 13]. This historical issue has been resolved and is completely unrelated to the present instances of IID, since dimer acid based additives are no longer widely used and current IID issues are observed in systems that are fully fuel lubricated in which no possibility of contamination by engine oil.

NORTH AMERICA - Many published studies of IID relate to North America, due in part to the proactive efforts of industry bodies in that market [1, 2]. Issues began shortly after the introduction of Ultra Low Sulphur Diesel (ULSD) in 2006. It has been suggested that such fuels may contain additives not commonly needed in higher sulphur blends. However, it is unlikely that North American IID is directly related to either decreasing sulphur or biodiesel usage. Evidence exists that North American IID is comprised of sodium salts of alkenyl (hexadecenyl or dodecenyl) succinic acids, likely formed by reaction of sodium with alkenyl succinic acids, which are widely used as pipeline corrosion inhibitors [1].

EUROPE - Soft soap like IIDs have been reported by a number of automotive manufacturers in the European market particularly in France and to a lesser extent Spain, Belgium and Germany. Sodium salt deposits were first observed around the time that biodiesel was introduced to the French market, as plotted in Figure 2. An identical relationship is observed in the Spanish market, where biodiesel concentration increased in about 2007.

Sodium hydroxide is used within the biodiesel production process and most biodiesel will contain sodium within the 5 mg/kg limit provided in the EN 14214 standard. However, it must be emphasized that such deposits do occur in FAME free fuels, which indicates that biodiesel is not the primary cause. Sodium may come from a range of sources, including: salt driers and caustic water used at refineries, sodium nitrite salts used as corrosion inhibitors, storage tank water bottoms and seawater used as ship ballast, bleaching agents (such as sodium hypochlorite), while acids may be present from a number of sources.

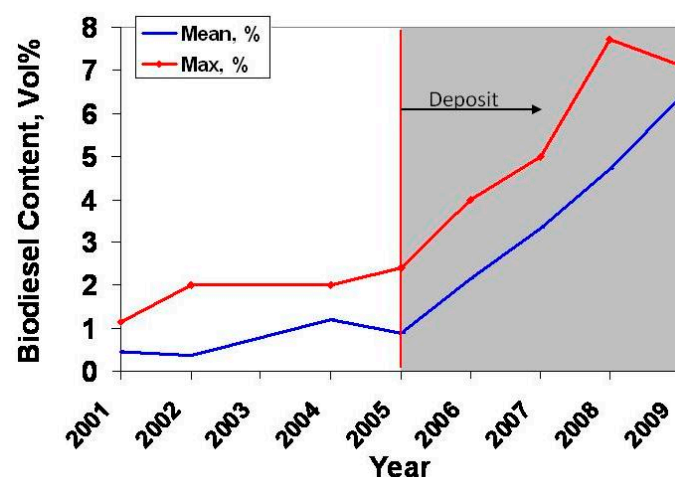


Figure 2 – Reports of IID as a Function of Biodiesel Usage in France

Figure 3 shows the fraction of all IID deposits that contain zinc, copper, sodium and chlorine, which are the most commonly observed elements after carbon and oxygen. As expected the majority of *nozzle-hole* coking deposits contain measurable zinc, while copper, sodium and chlorine are present in significantly less examples. In contrast, sodium is present in approximately 75% of examples of IID, while zinc is measured in only 35% of such deposit occurrences. Other elements such as potassium and calcium have also been observed and have the potential to create insoluble soaps. Previous publications indicate calcium may be less active than sodium [4], while little information is available for potassium.

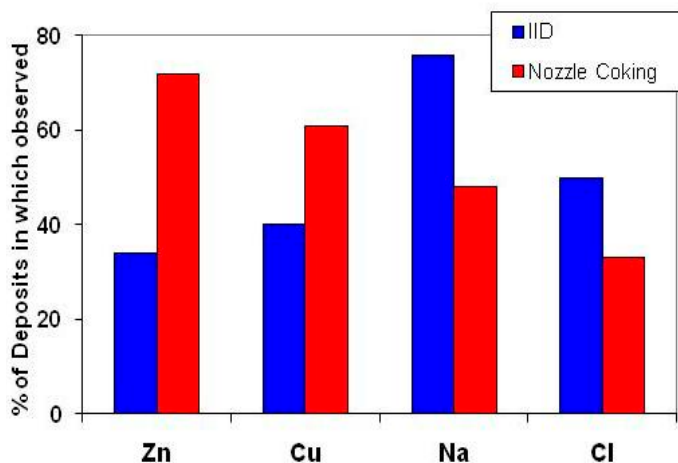


Figure 3 – Comparison of Elements Found in IID and Nozzle-hole Deposits

Fourier Transform Infrared (FTIR) spectroscopy was performed using a microscope-spectrometer PERKIN ELMER Spectrum Spotlight 200 and provided chemical structure information. The Infrared analysis was performed in the Attenuated Total Reflection mono-reflexion mode of the infrared microscope. A germanium crystal or a diamond plate was used depending on the deposit thickness. This technique allowed analysis of low deposit content (a few mg) without sample preparation. It can be used for liquid or solid samples, with an analysis area of $100 \times 100 \mu\text{m}$. Spectrum resolution is 4 cm^{-1} .

FTIR spectra showing the range of observed European IID are provided in Figure 4. The trace in blue represents a strongly soap based deposit with peaks at 1560 , 1438 and 1415 cm^{-1} indicative of a carboxylic acid salt. The presence of sodium in most IID indicates it is likely to be the main carboxylate salt created in practical operation. This is broadly similar to results of previous studies, which indicated the likely presence of sodium C18 fatty acid soap [5]. The presence of a substantial concentration of chlorine in some IID indicates that sodium chloride may be present in some fuel batches.

At the other extreme, the trace in red is from a highly polymeric deposit, with a strong peak at 1658 cm^{-1} indicating the presence of a carbonyl group from an amide or peptide bond. This peak combined with one at 1550 cm^{-1} matches with some polyisobutylene succinimide (PIBSI peaks), although the latter is sometimes obscured if carboxylates are present. The sensitivity limitations of the FTIR mean that PIBSI should not normally be visible on fuel wetted surfaces. Despite this, the infrared spectra of polymeric IID consistently show evidence of PIBSI, with peaks at 1471 , 1390 , 1366 and 1231 cm^{-1} assigned to PIB and at 1660 cm^{-1} to amide in this and previous studies [5].

PIBSI has been identified as a contributor to IID formation due to reaction with fatty acid based materials,

specifically di-fatty acid materials [4]. In practice, most European deposits are a combination of several influences and so show evidence of both soap and carbonyl deposits, as shown by the black line in Figure 4.

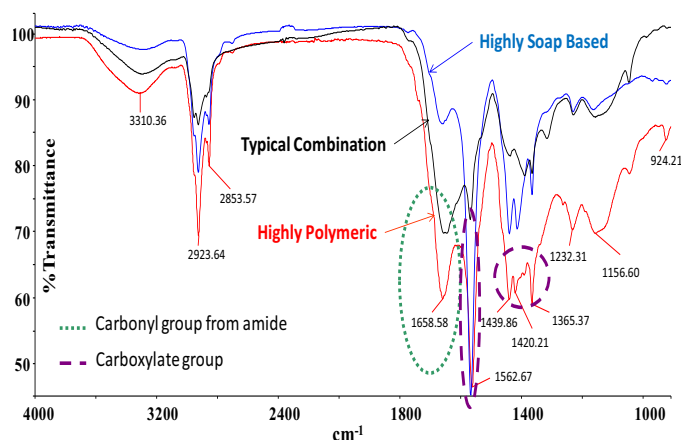


Figure 4 – FTIR Spectra of Typical IID from European Field Returns

A comparison of the spectra from deposits in North America and Europe is provided in Figure 5. The spectrum of North American deposit is taken from Reference 1 and is believed to have been formed by reaction of hexadecenyl succinic acid and sodium. Hexadecenyl succinic acid is part of the alkenyl succinic acid family used as a corrosion inhibitor.

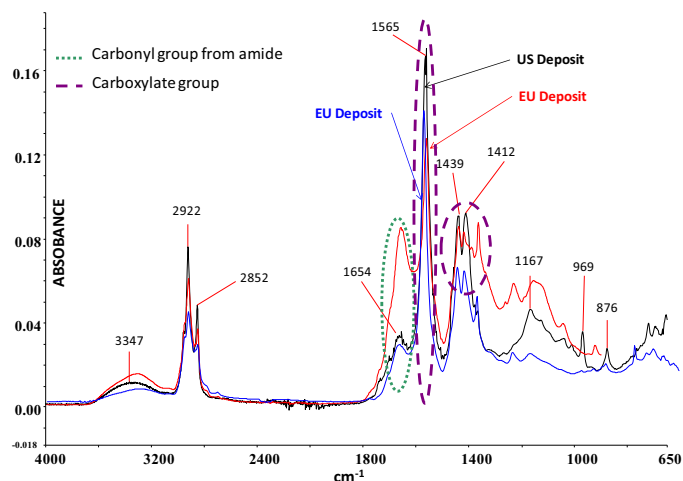


Figure 5 – Comparison of FTIR Spectra from IID in Europe and North America
(North American spectrum taken from Reference 1)

The North American spectrum is very similar to that of some European soap based deposit that exhibits a strong carboxylate trait with a large peak at 1564 cm^{-1} and a smaller peak around 1650 cm^{-1} (blue plot in Figure 5). However, information available to date indicates the North American deposits do not normally contain the peaks associated with PIB and amides, while the other European IID (red plot in figure 5) have a hard polymeric

structure, possibly due to the fact that PIBSI is not widely used in the US market.

FILTER DEPOSITS – Occasional incidence of filter plugging have also been reported [2, 14]. Two broad categories of deposit formation are observed on filter elements. Soft soap like deposit has been observed from a number of regions. These are typically of light colour and may occur within the fuel distribution system, which indicates they are not due to the aging within the FIE. Issues have been reported in warm regions such as Spain, which indicates that they are not due to precipitation of saturated monoglycerides or similar materials in FAME. Anecdotal evidence of hard dark filter deposit exists from the North American market and in applications where very severe fuel recirculation occurs. The two reported filter deposit mechanisms bear a superficial resemblance to those in IID; however no data exists to indicate a positive linkage.

IID TEST METHODOLOGY

IID TEST APPARATUS – In the past, deposits formed within the injector nozzle-holes have been difficult to simulate in a laboratory setting in the absence of a fired engine test. In contrast, IID's are continuously fuel wetted with no possibility of vaporization or exposure to the combustion gasses. As a result, it should be possible to simulate the deposit process in the absence of a fired engine, assuming that appropriate temperature conditions are achieved. It is likely that the deposits may be driven in part by fuel pressure, which in modern passenger car FIE may exceed 200MPa.

Such pressures are difficult to achieve in a laboratory setting and the present study uses FIE mounted on an electrically operated test stand. The apparatus shown schematically in Figure 6 was developed to replicate deposit formation under conditions similar to those found in real world operation, using a system design which recreates that found in a typical passenger car. An electric motor driven fuel injection pump provides pressurized fuel to a common rail feeding a single fuel injector. Since no combustion takes place the injected fuel is passed to a separate waste tank and no recirculation of normally combusted fuel occurs.

Return fuel from the high pressure pump, rail and injector is routed to the main storage tank, exactly as on an operating vehicle. The fuel injector is mounted in an electrically heated block, which replicates the heat flow to the injector tip from the combustion process in engine operation. Nitrogen gas is used to prevent auto ignition within the heated block, which serves as a catchment vessel for the injected fuel. No copper or zinc coated materials are used anywhere in the system. No filter is used in the system to reduce required fuel volume and also to minimize potential for cross contamination between tests.

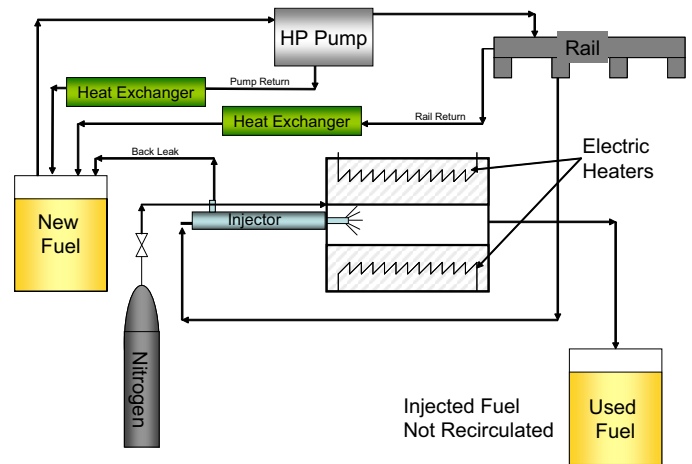


Figure 6 – Schematic Diagram of Internal Injector Deposit (IID) Apparatus

IID TEST CONDITIONS – The operating conditions used in the IID apparatus are summarized in Table 1 and were selected to simulate thermal soak-back to the injector that occurs following severe engine operation. Occasional operation under these conditions in practical operation is believed to result in gradual accumulation of deposit over the life of the system. Full-scale engine tests typically recreate such conditions using cyclical test conditions of alternating high and low load. As the present test does not use a fired engine there is the opportunity to continuously create a soak back environment by combining high fuel rail pressures and temperatures with a low injected volume. The IID test condenses a lifetime of occasional soak-back operation to produce measureable deposits with a relatively small fuel volume in a brief duration.

A rail pressure of 160 MPa was selected as typical of recent common rail applications and is lower than many current and future designs. The injection cycle was selected to produce a flow rate similar to that of a vehicle at idle, which results in a test volume of approximately 4 Liters during a 7.5 hour test from the single injector. The complete system is rinsed for 10 minutes with test fuel prior to operation to remove any residual fuel from previous tests.

A new injector is used for each test. The duration of injection is fine tuned during the first 30 minutes of operation to ensure constant flow rates between tests. Following each test the injector is disassembled and the deposit thickness measured using a Zygo Interferometer. This non contact method avoids possibility of inadvertent contamination of deposits intended for subsequent surface chemical analysis. It was not possible to accurately measure IID with a thickness below 0.5 microns, which approximates the surface finish of the components.

Measurements obtained using a thermocouple mounted close to the needle seat during fired multi-cylinder engine