

Biodiesel Feedstock and Contaminant Contributions to Diesel Fuel Filter Blocking

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ABSTRACT

In recent years, the number of complaints and the severity of premature diesel fuel filter plugging have increased dramatically in the U.S. and Europe. These instances are often accompanied by longer start up times, poor drivability, and increased maintenance across different fuel filter applications. The rise in these instances of filter plugging is closely associated with the increasing prevalence of high pressure common rail (HPCR) fuel systems and the growing usage of biodiesel. Smaller pore size restrictions for fuel filters due to tighter clearances in HPCR injectors, coupled with contaminants from biodiesel and carboxylate salts in fuel, have been identified as accelerants of diesel fuel filter plugging.

Testing protocols will be reported that can be used to screen contaminant-doped B10 fuels (10% FAME biodiesel in ULSD) to determine their propensity to plug fuel filters. Fuels were evaluated using bench-top filtration testing equipment to determine their filter blocking tendency (FBT) values. Three biodiesel related contaminants were investigated: saturated monoglycerides, sterol glucosides, and carboxylate salts.

Test results will show that the specific feedstock of biodiesel - from used cooking oil to coconut oil - can alter the filterability of biodiesel-containing fuels. The same contaminant level may or may not cause filter blocking depending on the biodiesel feedstock. Additionally, the treatment of contaminant-doped fuels with commercially available deposit control additives (DCAs) will be shown to prevent or mitigate diesel fuel filter blocking.

Important parameters for test fuel preparation will also be highlighted that could prove useful for internal diesel injector deposit (IDID) engine test development.

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INTRODUCTION

Mandates for lower pollutant emission levels in recent years have led to alterations in both engine design and diesel fuel composition. In order to meet these stricter emissions requirements for diesel engines, HPCR fuel systems have become more prevalent. HPCR injectors require very small clearances between internal moving parts in order to achieve the high pressures necessary for optimum performance. In order to protect the fuel systems, filter technology and pore sizes have changed. Fuel filter pore sizes have decreased in size to as low as 2-5µm in secondary filters (Figure 1). When filters perform their jobs of preventing particulate matter from entering the fuel injection systems, more material is being filtered out at an elevated rate leading to increasing instances of filter blocking.

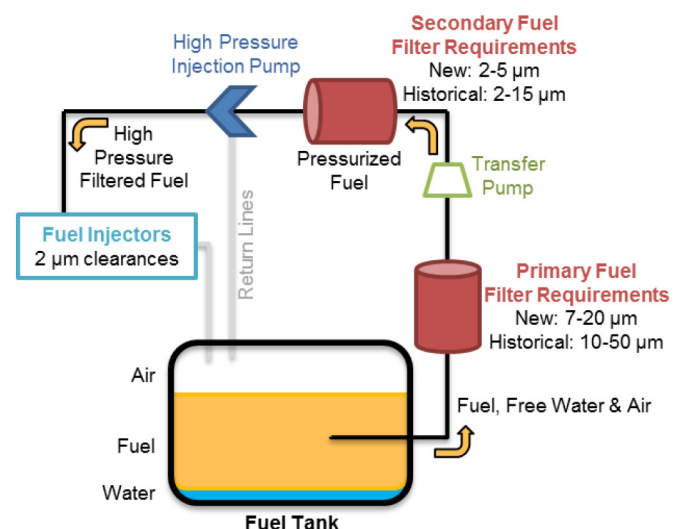


Figure 1. Diagram of an engine fuel filtration system designed to prevent hard particles from entering the fuel injection system. Red Documents
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cylinders represent filters that the fuel (yellow arrow) travels through on its way to the injectors.

As filters plug prematurely before typical maintenance cycles, new field complaints have arisen and others have become noticeably more prevalent. These complaints have included slow fuel flow rates, longer start up times, overall poor drivability, increased maintenance, and even complete engine shut off. Filter plugging has led to increased equipment costs, increased maintenance costs, decreased production, and very costly engine downtime. Additionally, fuel exposure to lower temperatures can exacerbate filter blocking issues leading to extra required maintenance.

To identify the cause of filter blocking in the field, more than 50 blocked filters from the U.S. were analyzed. The primary causes of the filter blocking are shown in [Figure 2](#). In many cases, combinations of filter blocking materials were identified. With this field information and industry reports of biodiesel sterol glucosides (SGs) causing filter blocking [1, 2], a test matrix was developed. Note: Moser [3] defines 'minor components' of biodiesel as naturally occurring species found in vegetable oils and animal fats, and biodiesel 'contaminants' as incomplete or unwanted biodiesel production reaction products. For this paper, the term contaminant refers to any non-fatty acid methyl ester species that contributes to filter blocking. This will encompass both naturally occurring species, such as sterol glucosides, as well as incomplete reaction products, namely saturated monoglycerides (SMGs).

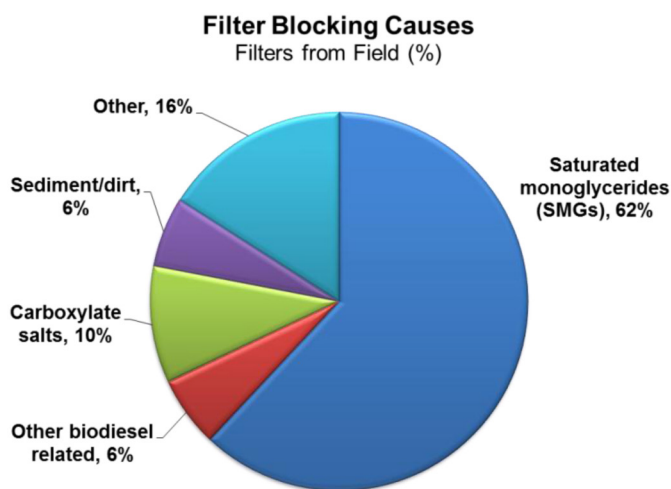


Figure 2. U.S. field fuel filter issues as identified by analysis at Afton laboratories.

There are several industry tests that evaluate fuel filtration behavior. These tests all measure the pressure as fuel is pumped through a specified filter membrane at a constant rate, but differ in the fuel blend type and test fuel preparation method. Existing tests include 1) the Standard Test Method for Determining Filter Blocking Tendency (FBT) (ASTM D2068 / IP 387) which is the general procedure used for middle distillate fuels including biodiesel and biodiesel blends; 2) the Canadian Cold Soak Filter Blocking Tendency test (CSFBT) which specifies testing B20 (20% by volume biodiesel in petrodiesel)

fuels soaked at 1°C for 16 hours; and 3) the European Cold Soak test (CS IP387) which provides guidelines for testing B10 blends that are soaked at 5 °C for 16 hours. The latter two tests evaluate how blending with petrodiesel fuel (B0) and cold exposure can affect overall fuel solvency. Another filtration test explicitly for biodiesel (B100) is the Cold Soak Filtration Test (CSFT, ASTM D7501) which assesses B100 after ~5 °C exposure for 16 hours using vacuum filtration.

While these tests all evaluate fuels in their native states, there is a need to test fuels that are intentionally contaminated with materials known to cause filter plugging. First, it is desirable to have a way to determine at what levels the problematic materials start to cause issues. Secondly, the tests could be used to study the effects of different base fuels and biodiesel on filterability. Lastly, the methods could be used to determine if commercially available deposit control additives (DCAs) are able to alleviate filter blocking. It should be noted that DCAs that pass the materials through the filter to avoid filter blocking must also be effective in preventing diesel injector deposit formation from these materials.

Biodiesel manufactured in the U.S. and Europe must pass requirements for amounts of free and total glycerin set by either ASTM standard D6751 Standard Specification for Biodiesel Fuel Blend Stock (B100) for Middle Distillate Fuels [4], or European Committee of Standards (CEN) standard EN14214 Fatty Acid Methyl Esters (FAME) for Diesel Engines, Requirements and Test Methods [5], as shown in [Table 1](#). It should be noted that the two test methods to quantify free and total glycerin (EN14105 and D6584) were developed around biodiesels such as soybean, rapeseed, and sunflower, and they are not always suitable for testing biodiesels made from lauric acid oils, such as coconut or palm kernel. Both the CEN and ASTM standards set maximum limits for total monoglycerides. Mono-, di-, and tri-glycerides are partially or unreacted oils that are often considered contaminants in finished biodiesel. Monoglycerides can start out being generally soluble in biodiesel, but cold temperatures often cause them to precipitate from solution and they will only dissolve back into solution at elevated temperatures [6]. Depending on the biodiesel feedstock and fatty acid distribution, a portion of the monoglycerides will be saturated (SMGs) which can vary from 7% for rapeseed methyl ester (RME), to 45% for palm methyl ester (PME). Due to their linear structure ([Appendix 1a](#)), SMGs are better able to pack together, and thus have a higher propensity to form solid precipitates.

Table 1. Free and total glycerin specifications for biodiesel as required by EN14214 (test method EN14105) and ASTM D6751 (test method D6584).

Species	Max limit by EN14214 (wt% : ppmw)	Max limit by ASTM D6751 (wt% : ppmw)
Free glycerin	0.02 : 200	0.020 : 200
Monoglycerides	0.80 : 8000	0.40 : 4000
Diglycerides	0.20 : 2000	NA
Triglycerides	0.20 : 2000	NA
Total glycerin	0.25 : 2500	0.240 : 2400



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Sterol glucosides (SGs) (also referred to as steryl glucosides, steryl glycosides, sterol glycosides, or phytosterols) (Appendix 1b) have been reported as another problematic biodiesel related species. SGs come from plant membranes and are soluble in vegetable oil in their original acylated state. The main issue arises in the biodiesel transesterification process when they become deacylated and subsequently lose solubility. While a portion of the SGs are removed in processing steps, finished biodiesel can still contain amounts that are capable of causing issues. SGs have historically been difficult to accurately quantify due to their low concentrations. However, new methods have been developed [7, 8, 9] to detect and quantify sterol glucosides. Biodiesel standards and specifications do not currently put limits on amounts of SGs allowed in biodiesel fuels.

METHODS AND MATERIALS

Filtration Test and Instrumentation

The published testing method Procedure B for ASTM D2068, Standard Method for Determining Filter Blocking Tendency [10], was followed for all of the fuels tested below. The method stipulates that the rig pressure is monitored as 300 mL of fuel is pumped through a 1.6 μm Whatman glass fiber filter at a rate of 20 mL/min. The test is completed once one of the following conditions is met: 1) the entire 300 mL of fuel is pumped through, or 2) the pressure reaches 105 kPa prior to all of the fuel flowing through the filter. If all the fuel makes it through the filter, the final pressure is recorded and Equation 1 is used to calculate the FBT value. Alternatively, if the 105 kPa pressure limit is reached before 300 mL of fuel is pumped through the filter, the pumped volume of fuel is recorded and Equation 2 is used to calculate the FBT value.

$$FBT = \sqrt{1 + \left(\frac{P}{105}\right)^2} \quad (1)$$

$$FBT = \sqrt{1 + \left(\frac{300}{V}\right)^2} \quad (2)$$

Equation 1. 'P' is the maximum pressure obtained in kPa. Values range between 1 and 1.41.

Equation 2. 'V' is the volume in mL of fuel passed through the filter before failure. Values range between 1.41 and 30.02.

Generally, FBT values of less than 1.41 represent fuels with good filtration properties, while fuels with values higher than 1.41 indicate that the fuel could have an increased tendency to block a filter and cause further issues. A reference table of the common FBT values is listed in Table 2. A Stanhope-Seta Multi Filtration Tester Model 91600-3 was used to measure the FBT of each fuel. The basics of the instrument are illustrated in Figure 3.

Table 2. FBT values correlated to the end of test conditions

	FBT Value	Volume thru filter (mL)	Pressure at end of test (kPa)
PASS	1.01	300	15
	1.04	300	30
	1.13	300	55
	1.26	300	80
	1.38	300	100
	1.41	300	105
FAIL	1.74	210	105
	2.69	120	105
	3.16	100	105
	4.40	70	105
	5.10	60	105
	6.08	50	105
	7.57	40	105
	10.05	30	105
	15.03	20	105
	30.02	10	105

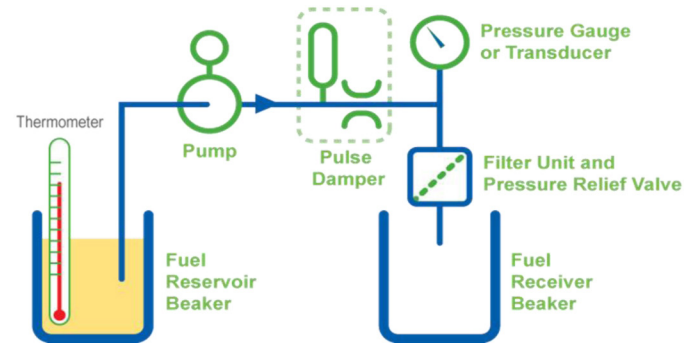


Figure 3. Schematic of the basic setup of the instrument used to measure FBT [10].

Test Reproducibility

The equation for reproducibility precision values stated in the D2068 test method was used to calculate data point standard deviations. These values appear in data tables as plus/minus values and in the graphs as error bars. These values were taken into account when determining whether or not two values were statistically different from one another. For example, considering the SME (soy methyl ester) data in Table 3, the listed FBT values for each of the samples from 500 to 1500 ppmw SMGs would all be considered equivalent.

Fuel Sample Preparations

Base Fuels

The base fuel (B0) used for all testing was a DF-79-07 reference diesel fuel. The B0 fuel was kept constant throughout the testing to ensure that the observed filter blocking effects were solely a result of the biodiesels and the added dopants. Four fatty acid methyl ester (FAME) biodiesels (B100) were used: a used cooking oil methyl ester (UCOME), a soy methyl ester (SME), a rapeseed methyl ester (RME), and a coconut methyl ester (CME). Prior to blending and testing, the B0 and B100 fuels were vacuum filtered through 1.6 μm Whatman glass fiber filters. The filters were made of the same material used in the syringe filters for the FBT test. This was to ensure that the only contaminants contributing to the filter blocking were the contaminants purposefully doped into the fuel.