

Glycerin Related Premature Plugging of Fuel Filters

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With the introduction of the Energy Policy Act of 2005, there has been an increasing usage of biodiesel across the United States. The growing usage of these renewable fuels has created several challenges related to filtration. Foremost of these challenges is the removal of residual glycerin from both neat biodiesel and biodiesel blends. Glycerin is a byproduct of the transesterification process used to treat biodiesel feedstocks and is partially removed from fuel through coalescence, centrifugation, and water washing. Over the last ten years Donaldson scientists have investigated several hundred fuel filters that exhibited premature plugging in applications of biodiesel filtration. These filters were from all major manufacturers, constructed of typical fiber materials (glass, cellulose, polyester), and were found to have been extensively fouled with insoluble free glycerin. In this report we detail that biodiesel fuels produced to meet or exceed current standards can still precipitate insoluble glycerin when cooled or when blended with ULSD. We also demonstrate that precipitated glycerin can cause significantly reduced life of fuel filters. Note that all references to glycerin in this report are specific to free glycerin and not alkyl glycerides.

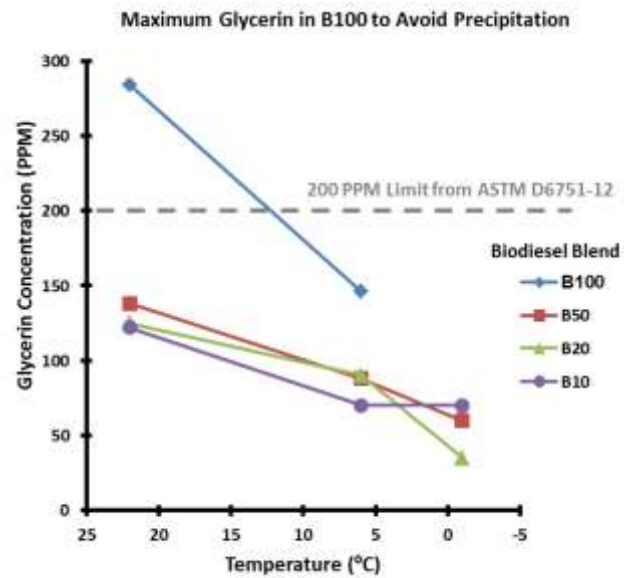
The standard that dictates the amount of glycerin in B100 biodiesel in the U.S. is ASTM D6751-12. This document limits the maximum allowable level of glycerin in B100 to 0.020% by weight (200 PPM). The analytical method specified to determine glycerin in the fuel is ASTM D6584, which involves derivitization of glycerin into its trimethylsilyl ester with N-Methyl-N-(trimethylsilyl)trifluoroacetamide and analysis with nonpolar column gas chromatography.

Applying ASTM D6584 to determine the equilibrium solubility of glycerin in a commercially available B100 at room temperature (22 °C) yields a result of 285 PPM, well above the allowable amount. However, when the temperature of the B100 is reduced to 6.5 °C, the maximum solubility drops to only 146 PPM. This reduction in solubility means that if biodiesel is produced containing maximum allowable amount of glycerin and cooled to 6.5 °C it will lose 56 PPM of glycerin to insoluble particles. In a 10,000 gallon tank of biodiesel, this is equivalent to precipitating nearly 4 pounds (0.38 gallons) of filter plugging glycerin particles!

Similarly problematic is the reduced solubility for glycerin when biodiesel is blended with ULSD. Glycerin is a relatively small polar molecule and its solubility in a fuel is heavily influenced by that fuel's polarity. Biodiesel is composed of fatty-acid methyl esters (FAME) and has relatively higher polarity compared to ULSD, which is primarily composed of paraffin compounds. In the following examples we demonstrate that blending biodiesel with ULSD decreases the polarity of the fuel blend and is accompanied by a decrease in glycerin solubility.

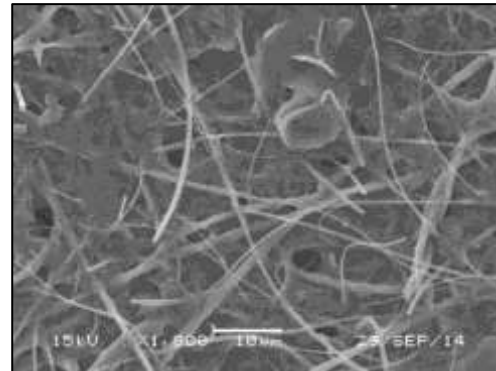
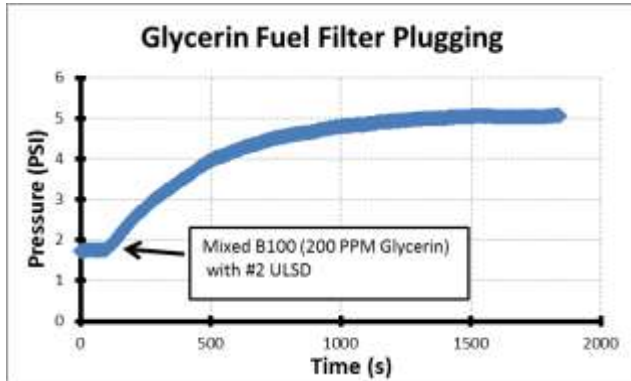
As shown in the table below, dilution of B100 with #2 ULSD creates a fuel with far lower glycerin solubility. For example, a B10 blend at room temperature was found to have a glycerin solubility limit of 12 PPM. In order to avoid precipitation, the initial B100 used to make the blend should contain no more than 120 PPM of glycerin. Similar to our example above, if a batch of biodiesel produced with the maximum allowable amount of glycerin and blended with ULSD to create B10, that blend will have lost at least 40% of its glycerin to insoluble particles. For a 10,000 gallon tanker of fuel, this equates to 0.5 pounds (3/4 cup) of insoluble glycerin that will plug modern fuel filters. In order to prevent glycerin related filter plugging when blending biodiesel, the initial B100 glycerin level must be below the value listed in the rightmost table column and shown in the following plot. From this data it is clear to see that the maximum allowable glycerin content of 200 PPM as outlined by ASTM D6751-12 is not adequate for biodiesel blends or B100 at reduced temperatures.

Sample (Temperature)	Solubility Limit (PPM)	Calculated of Maximum Glycerin in B100 (PPM)
B100 (22 °C)	284	285
B50 (22 °C)	69	138
B20 (22 °C)	25	125
B10 (22 °C)	12	120
B5 (22 °C)	5	100
B100 (6.5 °C)	146	146
B50 (6.5 °C)	44	88
B20 (6.5 °C)	18	90
B10 (6.5 °C)	7	70
B50 (-1 °C)	30	60
B20 (-1 °C)	8	40
B10 (-1 °C)	8	80



The effect of insoluble glycerin particles on high efficiency filter media was measured by creating B100 with the maximum allowable amount of free glycerin, blending with ULSD, and passing the fuel through a patch of filter media. In this experiment 1 liter of B100 containing 200 PPM free glycerin was mixed at a 1:1 ratio with #2 ULSD at room temperature. This solution

was passed through a piece of high efficiency filter media and the pressure across the filter was recorded. Recirculating 2 liters of the B50 solution at 0.7 feet/min through a 13.8 cm² filter patch produced a pressure increase of nearly 3.5 PSI (left plot). This amount of flow is equivalent to a full size filter element treating only 400 gallons of fuel. The pressure drop is clearly observed with electron microscopy (right image) to be from a glycerin film that developed on the filter media as it caught and removed glycerin particles. This image is typical of fuel filters found to be fouled with glycerin in the field.



In order to mitigate glycerin related fuel filter plugging Donaldson scientists recommend that the B100 fuel used to make biodiesel blends be further refined to bring the initial free glycerin level to an appropriate level. For most applications 50 PPM of free glycerin should be sufficient to avoid precipitation and premature filter plugging.