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Cold Soak Filtration Test

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A few years ago, the biodiesel industry faced an unusual problem. Fuel that passed all of the requirements of the ASTM specification would, on occasions, still plug fuel filters. This was unacceptable and the industry needed to know why and how to remedy the situation.

At the time the ASTM specification for biodiesel contained many tests that were intended to measure and limit the amounts of contaminants that could cause operational problems for biodiesel users. The specification had been used for about 10 years and was very successful in minimizing customer complaints when using the fuel. However, in cases when fuel that met all of the requirements of ASTM D 6751 but still plugged fuel filters, analysis of the filter residues and sediments in fuel tanks revealed that a variety of different compounds were responsible.

Some of the compounds found on the filters were not a surprise. Just like with petroleum-based diesel fuel, when water is present, microbes will grow at the water-fuel interface. The resulting biomass, if drawn into the fuel filter, is a known cause of plugging. Similarly, if pure biodiesel, or a high level blend of biodiesel, is stored in a tank that had previously held diesel fuel, the change in solvency of the fuel can loosen the accumulated deposits that can cause filter plugging. In cases where the ambient temperature is at or below the fuel's cloud point, the fuel itself could be solidifying and causing filters to plug. This problem frequently occurs when biodiesel is blended with a cold diesel fuel. All of these problems are now well-understood and their occurrence has been greatly reduced through education and strict adherence to handling and use guidelines [1].

Analysis of the residues from the plugged filters showed that two classes of compounds were found most frequently. These were monoglycerides formed from saturated fatty acids and sterol glucosides. Monoglycerides are intermediates of the biodiesel production reaction and their presence reflects a production reaction that was not complete. Saturated monoglycerides are a relatively small fraction of the total amount of monoglycerides (about 15% for soybean biodiesel). They have high melting points (about 176°F or 80°C) and if they are present in excessive amounts, they will crystallize and form sediments.

Sterol glucosides are complex molecules consisting of a glucose ring coupled to a sterol. These compounds are found naturally in many vegetable oils, including soybean and canola [2]. Sterol glucosides, also known as steryl glucosides, are soluble in oil but when the oil is transesterified to produce biodiesel, the sterol glucosides are converted to an insoluble form [3]. Because of their low concentration, these compounds crystallize from the biodiesel slowly and may take several days to form sand-like particles that readily plug fuel filters [3, 4]. These particles can combine with water, saturated mono-glycerides, and other contaminants to form particle complexes that multiply the effects of the individual contaminants themselves [3]. A photograph of sterol glucosides isolated from canola-based biodiesel is shown below.

Figure 1. Sterol Glucosides Isolated from Biodiesel.

The ASTM specification has no limit specified for sterol glucosides and monoglycerides are controlled indirectly through the total glycerin limit although no distinction is made for saturated compounds, which have much higher melting points. When these compounds precipitate from a solution (such as biodiesel), they have become known as "above the cloud point precipitates." [5]

It is important to note that these compounds are not necessarily associated with the cold flow properties of the fuel. A fuel with a low cloud point, such as canola-based biodiesel (-2°C or 28°F), may cause filters to plug even at temperatures of 21-27°C (70-80°F) if sterol glucosides or saturated monoglycerides are present.

Direct measurement of sterol glucosides and saturated monoglycerides is difficult and expensive [6] so the industry has opted for a performance-based test that characterizes the tendency of the contaminants to cause filter plugging. A simple procedure was developed that has become known as the Cold Soak Filtration Test (CSFT) and is now accepted by ASTM as D 7501 [7]. The basic equipment for this test is shown in Figure 2. A small disk filter is clamped between a receiving flask and a funnel. Vacuum is applied across the filter with a safety flask to protect the vacuum pump. This equipment is common and readily available in most fuel testing laboratories and is essential for any biodiesel producer.

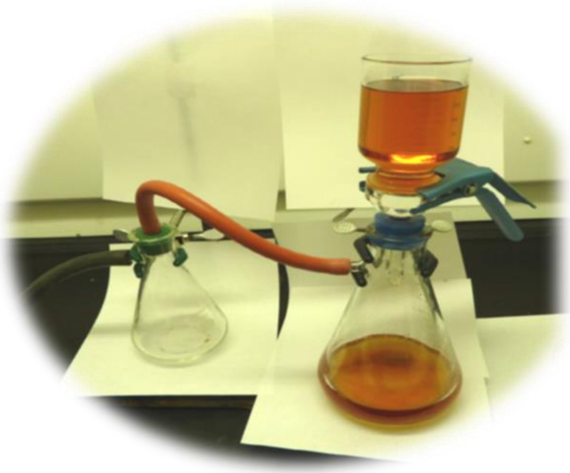

Figure 2. Cold Soak Filtration System. [7]

To conduct the CSFT, a sample of fuel is cooled to 4.5°C (40°F) for 16 hours to accelerate the formation of the precipitates. The length of time is critical because the molecular complexity and low concentration of the saturated monoglycerides and sterol glucosides make them very slow to crystallize. Then, the sample is heated to 25°C (77°F) to ensure that the methyl esters in the fuel itself are liquid so the sample will be filterable. Contaminants such as sterol glucosides and saturated monoglycerides have high melting points and are relatively insoluble so once they have crystallized, they remain solid at this temperature.

A 300 ml fuel sample is poured into the funnel and drawn through the filter with a vacuum of 70-85 kPa (21-25 in-Hg). The time for the fuel to pass through the filter is measured which must be less than 360 seconds for the fuel to be considered as passing. A premium grade of biodiesel, called 1-B, corresponds to a filtration time of 200 seconds or less and a concentration of mono-glycerides of 0.4% (by mass) or less.

ASTM D 7501 is conducted on B100 but most biodiesel is blended with diesel fuel. Diesel fuel is less polar than biodiesel. Since sterol glucosides and saturated monoglycerides are both polar, the solubility of these compounds could be expected to decrease as the biodiesel is blended with diesel fuel [5]. Thus, precipitation will be more likely after blending and re-dissolving of crystals less likely, even at higher temperatures.

The implementation of the CSFT has greatly reduced the frequency of filter plugging incidents caused by fuel quality. It should be noted that the CSFT does not identify the type or quantity of the contaminant that is causing the filter plugging. When a fuel producer has a fuel that does not pass the CSFT, the test results provide very little information that can be used to identify the source of the problem or how to resolve it. But as an inexpensive test that can be run by any producer, this test provides a rigorous check on commercial fuel quality and it has been a great success.

References

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