BIOdiesel TechNOTES

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ASTM BIODIESEL LABORATORY TESTS EXPLAINED

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This article follows TechNote #29A and explains what the biodiesel ASTM standards tests reveal.

ACID NUMBER

The acid number is a direct measure of free fatty acids (FFA), which can lead to corrosion and may be a symptom of water in the fuel. The acid value may increase with time as the fuel degrades due to contact with air or water. This test easy, inexpensive, and should be performed regularly as a part of the producer Quality Control (QC) program.

WATER AND SEDIMENT

Removal of all water is particularly important for biodiesel because it can degrade the fuel and supports microbial growth in storage tanks. The process should include steps to ensure the final product is dry and is kept dry. Suspended water is a problem because it contributes to the corrosion of the close-fitting parts in the fuel injection system.

Sediments may plug fuel filters and promote the formation of deposits on fuel injectors and other engine parts. Sediment levels in biodiesel may increase over time as the fuel degrades during extended storage. The production QC lab should be equipped to perform this centrifuge test on a routine basis.

FREE & TOTAL GLYCERIN & MONOGLYCERIDES

Elevated total glycerin values are indicators of an incomplete reaction and predictors of excessive carbon deposits in the engine and could lead to filter plugging. Bond glycerin is the glycerin portion of the mono-, di-, and triglyceride molecules and total glycerin is the sum of free and bond glycerin. Monoglycerides are generally considered the most harmful in biodiesel and can adversely affect cold flow properties. The glycerin tests are by far the most important and likely the most difficult to run in terms of accuracy. Glycerin tests require a gas chromatograph (CG) and the pains-taking calibrations and meticulous weights and measures that go along with it. The test for free and total glycerin must be done routinely as the key measurement in the producer's QC program.

CLOUD POINT

Of major concern in cold climates, the cloud point (CP) indicates the temperature above which an engine can operate without fear of plugging a filter and stranding the vehicle. It is highly dependent on feedstock, ranging from -8-15°C (18-59°) for 100-percent biodiesel. Highly unsaturated oils make for low CP biodiesel where highly saturated and hydrogenated fats and oils yield higher CP biodiesel. The producer can modify the cloud point with the use of additives or by blending feedstocks that are relatively high in saturated fatty acids with feedstocks that have a lower saturated fatty acid content. Producers are advised to be equipped to determine this property.

ALCOHOL CONTENT (one of the following must be met)

Flash Point

The flash point determines the flammability classification of material being tested. The typical flash point of pure biodiesel is above 200°C, classifying it as "non-flammable". However, during production and purification of biodiesel, if all of the methanol is not removed the fuel will be flammable and more dangerous to handle and store. Excess methanol in the fuel may also affect engine seals and elastomers and corrode metal components. Generally, a production QC laboratory should include a flash point apparatus for quality control and as a means of detecting excess alcohol levels.

or

Methanol Content

Methanol content of biodiesel fuel is an important factor in determining the tendency of the fuel to exhibit flammable characteristics. Methanol content is determined by a GC and one GC is generally calibrated for determining free and total glycerol. If biodiesel passes the flash point test at 130°C or greater, the methanol content test is not necessary.

SULFUR

Off-road fuel can have up to 500 parts per million (0.05%) sulfur and on-road fuel can only have 15 parts per million (0.0015%) of sulfur. Biodiesel feedstocks typically have little sulfur, but this test is an indicator of contamination of protein material, carryover catalyst or neutralization material from the production process. Producers using rendered or waste feedstocks should have access to this measurement since some of this material has been found to have sulfur levels of 40-50 ppm. Producers that do not meet this specification on a regular basis need to consider sulfur removal technology in their facility.

OXIDATION STABILITY
All stored fuels are subject to degradation over time. This degradation may be due to microbial action, water intrusion, or oxidation. This test determines how quickly it oxidizes. If it fails this test, the fuel can go bad too fast while in storage. There are several additives on the market that can help improve oxidative stability. Characteristics such as the acid number, water and sediment, and viscosity will change during storage and can generally be used to determine if the fuel has deteriorated. Should an aged fuel fail any of these three standards, it should not be used.

COLD SOAK FILTRATION
ASTM required this test after reports that biodiesel could form precipitates above the cloud point which can clog filters. The cold soak filtration test determines if biodiesel shows precipitate formation upon cooling to temperatures above the cloud point.

PHOSPHORUS
Phosphorus can come from incomplete refining of the phospholipids (or gums) from the vegetable oil and from bone and proteins encountered in the rendering process. The producer should have access to this method for periodic measurements.

SULFATED ASH
Sulfated ash is a measure of ash formed from inorganic metallic compounds. The ash-forming inorganic materials may be present in biodiesel from unremoved catalysts, abrasive solids, or soluble metallic soaps. Metal-containing fuel additives and catalysts are the main contributors of sulfated ash. Abrasive solids contribute to injector, fuel pump, piston and ring wear, and engine deposits. Soluble metallic soaps have little effect on wear but may contribute to filter plugging and engine deposits. The damage caused by ash from unburned hydrocarbon is different from the damage caused by metallic ash. It is abrasive and can cause serious damage to the interface between the piston ring and the cylinder wall.

CALCIUM & MAGNESIUM
Small levels of calcium and magnesium may collect in exhaust particulate removal devices and act as abrasive solids or soluble metallic soaps, which impacts are described above. The presence of these metals also contributes to the sulfated ash measurement. Calcium and magnesium can be introduced during the washing process due to the use of hard water.

SODIUM & POTASSIUM
Sodium and potassium, like calcium and magnesium, may be present in biodiesel as abrasive solids or soluble metallic soaps. This specification was included for the same reason as calcium and magnesium were. Sodium and potassium in biodiesel usually comes from the catalyst not being removed after the reaction. Use of catalyst in excess may result in having an off-spec biodiesel. If water and/or FFA is present during biodiesel reaction, some soap is formed and usually remains dissolved in biodiesel. These soaps can become sodium or potassium salts during neutralization.

KINEMATIC VISCOSITY
Kinematic viscosity is the resistance to flow of a fluid under gravity and fuel injectors will not perform properly if it is too high. The viscosity of biodiesel can be predicted (±15-percent) using the esters composition determined using a GC. The viscosity apparatus to run D 445 is not critical to the QC lab, but it is valuable as a quick method for estimating the degree of reaction completion.

COPPER STRIP CORROSION
This test monitors the presence of acids in the fuel. The most likely source of a test failure would be excessive free fatty acids, which are determined in accordance with the acid number. The producer may choose to run this test periodically, but the acid number (D 664) determination is the more important measurement.

CETANE
The cetane number is a measure of how easily the fuel will ignite in the engine. It is seldom an issue with biodiesel because all of the common fatty acid esters have cetane numbers near or above the minimum requirement. The cetane number can be predicted (±10-percent) using the esters composition. It is unlikely that an individual producer will ever run cetane tests on-site because the equipment is extremely expensive.

CARBON RESIDUE
The carbon residue is a measure of how much residual carbon remains after combustion. Carbon residues may decompose and pyrolyze to hard deposits and clog the fuel injectors. The most common cause of excess carbon residues in biodiesel is an excessive level of total glycerin. Total glycerin is also measured directly so this measurement is generally not critical.

DISTILLATION TEMPERATURE
According to the standards, this specification was incorporated as an added precaution to ensure the fuel has not been contaminated with high boiling contaminants. The high boiling vegetable oils are a result of certain long chain fatty acids. The ester composition allows calculation of the T-90 point without having to perform the testing for every batch of product. It is unlikely that the producer would have a reason to run this test except to certify compliance with the standard.