BIOdiesel Tech NOTES

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BIODIESEL LABORATORY TESTS AND EQUIPMENT

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When making biodiesel there is no more important factor than producing a quality product. In order for a diesel engine to function properly, the fuel needs to be laboratory tested before use.

Depending on the types of feedstock, process, and degree of processing experience, product quality can vary substantially. It is important to test and monitor the quality of the feedstock. Testing feedstock can usually be limited to water content and acid value. The acid number determines the amount of free fatty acids (FFA) in the feedstock, which helps producers adjust the catalyst to improve the yield for a successful batch of biodiesel. Excessive water and FFAs are the most common issues that producers have with their feedstock. Water will result in serious processing complications and feedstocks with high FFA levels must be dealt with by pretreatment or adjustments to the production process.

One of the first actions of the biodiesel industry was to develop a quality standard for biodiesel. Known as "The Biodiesel Standard", this was created under the auspices of the American Society of Testing Materials (ASTM D6751, Standard Specification for Biodiesel Fuel Blend Stock (100) for Middle Distillate Fuels). The purpose is to provide a biodiesel product that meets the requirements for blending with diesel fuel in a manner that will not adversely affect engine operation or drivability. The complete standard can be purchased from ASTM and further information on the standard is available on the ASTM website (www.astm.org).

The tests to ensure the quality of biodiesel require expensive equipment, and a small commercial facility requires the same quality assurance testing as a large one. Even noncommerical biodiesel producers need to perform an acid number test in order to determine the correct recipe prior to producing each batch of biodiesel. In most situations, an analytical laboratory capable of performing many of the tests is necessary on-site. It may be possible to commission some tests to a nearby commercial testing laboratory although that can also become expensive and the time lapse in getting the results could seriously disrupt the operation of the plant.

The table on the following page provides an overview of the testing requirements, the equipment needed to run the tests, and the approximate costs for the equipment. The table has been divided into three groups. The first group consists of those properties of the standard that can change from batch to batch and are essential. In the second group, there is a longer test interval for properties that may only need to be tested occasionally. Properties in the third group are not likely to change, are rarely or never in non-compliance, or can be reasonably predicted using other means. An explaination of each ASTM biodiesel test is provided in TechNote 28B.

Tests other than those required by the ASTM standards can be useful when making biodiesel, or may be more suitable or appropriate. The water and sediment test, for example, is performed by spinning a sample in a centrifuge at high speed. The main drawback to this method is that it lacks the capability to test bound water in the oil phase. Testing by the Karl Fischer titration method is a more accurate representation of all water in the fuel and is generally the preferred test at commercial operations.

There are also non-standard tests that can give an indication of several fuel characteristics, which are simple, cost-effective, and can be performed with a fast turnaround time. One such test is called the 27/3 test. The most important issue during biodiesel production is the completeness of the reaction. The key test to determine the completeness of the reaction employs a gas chromatograph (GC), which measures the glycerides and free glycerin, but GC's are expensive, require trained personnel to operate, and need frequent maintenance. A crude method to evaluate glycerides in biodiesel without a GC is the 27/3 test. The premise of the test is that methyl esters (biodiesel) are soluble in methanol while mono, di and triglycerides are not. The test simply dissolves a biodiesel sample into methanol. If there are no oil or fat globules visible after the biodiesel is dissolved into the methanol, the reaction has been adequate. Otherwise, a second reaction is needed. The test does not, however, measure the free glycerin since free glycerin is soluble in methanol.

Consistently making a fuel of good quality is essential. When making biodiesel, as with any other product, "Quality" must come first. For commercial biodiesel production, all ASTM tests must be done to certify compliance, and the ability to demonstrate that the fuel meets the standard requirements should not be underestimated.

Biodiesel Laboratory Tests and Approximate Equipment Costs

ASTM D 6751-15, "Standard Specification for Biodiesel Fuel Blend Stock (B100) for Middle Distillate Fuels," ASTM International

PROPERTY	LIMITS	METHOD	APPARATUS	APPROX. COST
GROUP 1				
Acid Number	0.50 mg KOH/g Max.	D 664	General Lab Equipment (Burette)	250
Water & Sediment	0.050% Vol. Max.	D 2709	Benchtop Centrifuge	6,600
Free Glycerin	0.020% Mass Max.	D 6584	Gas Chromatograph	18,000
Monoglycerides	0.4% Mass by wt Max	D6584	Gas Chromatograph	-
Total Glycerin	0.024% Mass Max.	D 6584	Gas Chromatograph	-
Cloud Point	Report ºC to Customer	D 2500	Cloud and Pour Point Bath	3,700
Alcohol Control (one of the following must be met)				
1. Methanol Content	0.2% Vol. Max.	EN 14110	Gas Chromatograph	-
2. Flash Point	130ºC Min	D 93	Koehler Pensky-Martens Flash Cup Tester	2,200
Sulfur – S 15 Grade	15 ppm Max.	D 5453	Ultraviolet Florescence	22,000
Sulfur – S 500 Grade	500 ppm Max.	D 5453		
Oxidation Stability	3 hours	EN 15751	Metrohm Rancimat Model 873	15,000
Cold Soak Filtration	200/360 Seconds Max.	D 7501	Vacuum Filtration System	2,500
GROUP 2				
Phosphorous	0.001% Mass Max.	D 4951	ICP Spectrophotometer	30,000
Sulfated Ash	0.020% Mass Max.	D 874	Isotemp Basic Muffle Furnace 0.58 cu ft.	2,500
Calcium & Magnesium	5 ppm Max.	EN 14538	ICP Spectrophotometer	
Sodium/Potassium	5 ppm Max.	D 5453	Ultraviolet Florescence	
Kinematic Viscosity	1.9-6.0 mm^2/sec. Max.	D 445	Constant Temperature Bath	3,400
			Viscometers, #75 1.6-8 cSt, #100 3-15 cSt	200
GROUP 3				
Copper Strip	No. 3 Max	D 130	Koehler K25330 Copper Strip Test Bath	3,700
Cetane	47 Min	D 613	Send to outside lab	
Carbon Residue	0.050% Mass Max.	D 4530	Coking oven and glassware	2,500
Distillation T90 AET	360 ºC Max	D 1160	Vacuum Distillation Unit	15,000
			Total Cost	$105,500