Biofuels analysis was once a time-consuming process. Now with the emergence of portable and handheld devices, results can come fast in the palm of producers' hands

Taking matters into hand

by Luke Buxton

Biofuels must be up to scratch as contaminated or improperly blended fuel can damage a company's bottom line.

To ensure product quality several options exist including laboratory analysis, field test kits and portable lab instruments.

Laboratory testing would be the ideal solution if everyone had the budget and the time to wait for the results.

But in a market where time is money, quick and efficient portable systems are now finding a fixed place in the market.

InfraCal Blend Analysers

On the move

In today's energy-hungry world fuel finds its way through miles of fixed infrastructure, and as it makes its way through pipeline terminal systems the risk of contamination further down the line is too great for operators to take. This is one place where portable equipment is ideal.

Pipelines are generally the most economical way to move fuel. The cost to ship fuel by rail is about five times more than by pipeline. By barge it is around two and a half times more and by truck it can be close to 20 times more. Several pipeline companies in the US are currently preparing for the inevitable increase of biofuels and have performed trial runs of both biodiesel blends and pure ethanol.

Ethanol is widely transported via pipeline by the likes of Kinder Morgan, and in Brazil oil producer Petrobras secured a preliminary environmental license in July to construct an ethanol pipeline. Magellan Midstream Partners and the US' largest ethanol producer Poet have also signed a massive $4 billion (€3.03 billion) deal to pipe ethanol 1,800 miles.

As for biodiesel, which is already transported via pipeline in Europe, Kinder Morgan Energy Partners announced in July 2009 that it had undertaken the first successful commercial shipment of biodiesel (B5) through a pipeline in the US.

Tests on the biodiesel blend were done with US-based Wilks Enterprise's mid-infrared analysers at the beginning and end of the pipeline to ensure the biodiesel met blend specifications.

The ASTM Method D 975 currently allows up to 5% biodiesel in diesel without the requirement for biodiesel content labelling. This can lead to contamination worries for those also transporting jet fuel.

Portable infrared analysis can easily verify that the fuel contains 500 ppm or less of biodiesel – assuring the operator that the fuel is not a FAME blend and safe to ship on its pipeline. Ethanol does not pose the same potential risks for jet fuel contamination but ethanol is water soluble and can absorb moisture that may accumulate in a pipeline which makes it unusable as a fuel. Water in ethanol is another test that can be performed by infrared.

For barge, rail or truck deliveries, the distributor benefits from having a quick analysis method to verify the load has the correct blend and the receiver can also make sure they have they have the correct delivery.

Mobile solutions

Austria-based analysis provider Grabner has sold analysis equipment to pipeline terminals, especially small ones with limited space where small portable instruments have advantages. In this field its Irox fuel analyser is especially useful because users are scanning for lots of different fuel properties in one, meaning the user does not need three different analysers to measure three different fuel properties.

Free methanol in traces needs to be completely removed, as this has a dramatic affect on flash point. Pure biodiesel has a flashpoint usually around 160°C. Only 0.1% methanol will lower this flash point to drop below 120°C. If there is more methanol left in biodiesel the flash point can easily drop below 60°C, which is a serious risk for transporting biodiesel.

One of the key advantages of Grabner instruments is that it uses a limited amount of sample. Irox can conduct fuel analysis with 15ml: most of this is used for rinsing. The advantage of portable analysis is it uses the subsequent sample for rinsing and flushing out the previous sample and takes a little amount for testing.

The Irox 2000 takes a sample of 13ml. To test vapour pressure it uses 10ml; for testing flash point the sample needed is smaller at 1-2ml.

The company's portable Irox 2000 ethanol/petrol analyser with Mid-FTIR (Fourier transform infrared) and the Irox Diesel were both first used as laboratory bench-top equipment to develop methods. The idea of the instrument was to give the market something which is push-button operated, so users do not need scientifically trained personnel to operate.

The machines are portable

and form the focal point of what is essentially a mobile lab. The Irox with solid cast spectrometer inside weighs 15kg.

Truck or tank

In the tank or at the truck loading terminal the two blending methods most commonly used are splash and in-line blending. Splash blending is usually used only for biodiesel. The diesel fuel and B100 are pumped separately into a delivery truck or storage tank. It is assumed the blend will be adequately mixed in the tank or by the time the truck arrives at the delivery site. However, at a demonstration test conducted with a Wilks InfraCal Biodiesel Blend Analyser five minutes after filling the truck for B20, a sample taken from the top measured 11.9% biodiesel while another from the bottom was 24.1%. Therefore if the first delivery of fuel for a splash blend loaded truck is only a few miles away on a smooth road, the chance for delivering an accurate blend/mix is far from guaranteed. And subsequent deliveries will also have an incorrect blend.

In-line (injection) blending offers better consistency. While this is typically used for ethanol, it is becoming more common for biodiesel. The biofuel is mixed as it is metered into the pipe with the diesel or petrol. Additional mixing occurs as the fuels enter the receiving tank or truck. For biodiesel, density and viscosity changes require adjustments to the meters for an accurate blend.

While ethanol does not have density or viscosity issues, off-spec product can be a costly error if it gets to the consumer at a fuel pump. Because ethanol is an excellent solvent, with engines that are not designed for levels higher than 10%, components can disintegrate and clog fuel filters, carburettor jets and injectors and cause a fuel related break down.

The Oxford RF Sensors handheld sensor is one of only a few on the market

A handy device

Biofuels seem to have evolved from first to second and even, arguably, third generation fuels. The technology to analyse biofuels appears to echo a similar evolutionary trend – from lab-based, to portable and now handheld.

There are only a handful of companies which supply handheld biofuels analysers.

UK-based Oxford Sensors' move to a handheld sensor was client driven. A potential client approached Oxford RF Sensors in 2009 and asked if it could measure biodiesel in diesel in a handheld fashion.

The client in question needed to test the diesel fuel at its network of commercial vehicle service centres. The client used lab-based FTIR and experienced delays of up to two days in receiving the results.

'The sensor measures for contamination, content and the amount of biofuel in the sample. If analysis involves a lab kit, often technicians must interpret lines and compare them on a chart and deduce if it is likely to be petrol, or water. It needs interpretation and skill,' Ross Walker, CEO of Oxford RF Sensors, says.

The handheld device works with two parameters around raw data. The first is the frequency of oscillation. The other parameter is amplitude, or the lossiness of fluid. The handheld device uses these two parameters to measure both the amount of biofuel in diesel and also the source of the biofuel and whether or not there is contamination.

Oxford RF Sensors' handheld device operates in the area of the electromagnetic spectrum, a longer wavelength, with lower frequency than infrared. It uses a technique called a marginal oscillator which is sensitive to change and robust enough that it will accommodate change. The majority of oscillators oscillate at fixed frequency. Oscillation can be sensitive to change, as it will stop oscillating. Conditions for maintaining instability are such if it is sensitive it stops, which can be used to check change. 'We are able to accommodate large electromagnetic changes in fluid and maintain oscillation but detect small changes of electromagnetic properties of the fluid. That was what we span out of the University of Oxford to do. It has not been done before.'

There are other handheld options on the market using infrared (IR) technology – US-based Paradigm Sensors' i-spec Q-100 biodiesel analyser costs $5,000 (€3,800) and Spectro's Spectro Fluid Scan Q1000 costs $19,000. Both use consumables: things like sample containers that must be used for each measurement and hence there is a cost per measurement. In the case of the $5,000 device there is a tiny sample cell that costs $30 each. Take 100 measurements and costs mount up to $3,000.

The Oxford RF Sensor handheld device costs €1,500, and it is a quick and simple, plug and play solution. Once the sensor is dipped into the sample the operator presses a button and gets the answer.

The other methods take several minutes. 'Our sensors are affordable; payback is almost instantaneous in moments as it diagnoses potential warranty claims, justifying its existence or prevents a

Setting standards

One main issue at the moment is there is a discrepancy between standards for biofuels and biofuels blends. The American National Standards Institute (ANSI) has undertaken an initiative to collect standards and have one common standard. Standards define what kind of test companies have to do for checking biofuels. The key markets at the moment are: composition, flash point, distillation and vapour pressure, mainly for a petrol/ethanol blend.

Oxford RF Sensors is pushing to get its handheld device adopted as a reference standard for biofuels analysis. First the device has to be tested, which will take time. 'If you look at reference standards they are all lab-based chemical or optical methods that sample and analyse. Were the Oxford RF Sensors handheld device to be adopted it would be the first in-field device to serve as a standard,' Ross Walker, CEO of Oxford RF Sensors, notes.

Standards define what kind of test companies have to do for checking biofuels and biofuel blends. Commodity inspection must follow standards for fuel quality inspection. Grabner Instruments works closely with the ASTM D.02 committee, which publishes international standards for petroleum testing. 'International standards for petroleum testing are essential to guarantee that the fuel meets quality requirements no matter where it is produced. Grabner usually develops new small-scale methods and thus sets new standards, which are accepted by all major standardisation organisations,' Oliver Sauer, director of marketing and sales at Grabner Instruments, says.

breakdown of vehicles.'

Oxford RF Sensors uses FTIR as a means to validate the results of its handheld sensor. Independent tests show the handheld device is as accurate as a £15,000 (€18,000) FTIR machine.

FTIR looks at the absorption of IR, and translates that into a signal where there are absorption bands: from the height of banks in the frequency spectrum one can interpret the data.

Burst bubble?

Despite the advantages many producers are not in a position to buy new equipment. 'A big used instruments market developed after US biodiesel producers filed for bankruptcy. With so many cheap used analyzers for sale, the need to buy new equipment decreased heavily, which is why we currently are not focusing on this market,' explains Oliver Sauer, director of marketing and sales at Grabner Instruments.

Price can also be prohibitive. The cost of Grabner Instruments´ analysers varies: the Irox spectrometer is more expensive, whereas standard analysers like vapour pressure and flash point testers are less expensive. The average price in central Europe on its biofuels testing instruments is €15-30,000.

UK-based Analytik found a similar problem with its LabSpec portable analyser.

'We found a great deal of interest, but nobody that was prepared to invest,' Analytik's Ian Laidlaw remarks. 'For that reason we have not particularly targeted the biofuel industry since and have never yet sold an instrument for that application, although the possibility remains open.'

Laidlaw goes on to say the main problem is that near infrared (NIR) is essentially a secondary technique. It is potentially very powerful as if calibrated correctly the end user could simply dip a probe into his sample, press one button and get a whole suite of analytical results within a matter of a few seconds.

However, the generation of appropriate calibration models seems to be a major issue because that requires access to a huge amount of samples and primary analysis data and it needs considerable scientific expertise. Further problems are access to sufficient samples and data (potentially very expensive and time consuming to get either when starting from scratch); high cost of instrumentation (a LabSpec5000 system could cost £40-50,000); and limited regulation within the industry currently.

The testing world is a tale of two sides. On the one hand new technology is being developed which can save producers time and money. Yet on the other hand second-hand technology exists which companies can pick up for less expense, negating the drive to try out, and potentially benefit from the new equipment. ●

For more information:
Oxford RF Sensors:
www.oxfordrfsensors.com
Grabner Instruments:
www.grabner-instruments.com
Wilks Enterprise:
www.WilksIR.com
Analytik:
www.analytik.co.uk

BioMCN produces and sells high quality 2nd generation bio-methanol. Our unique and innovative process converts crude glycerine into bio-methanol, thus using its full energy potential and achieving 70% CO_2 emission reduction. Bio-methanol is an EU recognized 2nd generation biofuel, which is entitled to be counted twice in accordance with the Renewable Energy Directive.

Our bio-methanol offers petrochemical companies an attractive and cost competitive solution in their quest for more sustainable 2nd generation biofuels. Bio-methanol is an extremely versatile product, either as a biofuel feedstock for bio-MTBE, biodiesel and bio-hydrogen or as a fuel in its own right (M85). And, because bio-methanol is chemically identical to regular methanol, it is perfectly suited to produce green alternatives of existing applications for the construction and automotive industry as well. BioMCN is making a substantial contribution to the development and use of better biofuels. Bio-methanol dramatically reduces green-house gas emissions, and avoids the social and environmental issues sometimes associated with biofuels.

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