

Inspection and quality control of biodiesel to meet EU standards

The varied characteristics of an increasing range of feedstocks for biodiesel production demand rigorous testing and inspection throughout the supply chain

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The European quality standard for biodiesel, EN 14214, is five years old. It was originally developed as a specification for biodiesel derived from rapeseed oils. But, as we know, today's biodiesel market is based on fuels produced from a variety of feedstocks. This has introduced some extra complexity to the need to meet the EU's biodiesel quality standards.

The manufacture and import into Europe of biodiesels based on palm and soya oil from the US, Latin America and Asia requires onward blending to achieve biodiesel product that conforms to the EN 14214 specification. Many feedstocks are available on today's market to produce biodiesel, including the more familiar rapeseed oil (RME), palm oil (PME), soya oil (SME) and canola oil (CME), as well as waste oils, tallow and newer feedstocks such as oils derived from algae and jatropha. The economics of the industry and external issues such as perceived conflict with food markets mean that biodiesel refineries using multiple feedstocks and blending activities involving biodiesels from multiple sources are increasingly common in Europe.

It is important to remember that the origin of the feedstock will always be linked to the final quality of the biodiesel derived from each feedstock. The critical parameters that must be tested to meet EN 14214 can be identified as cold flow properties, iodine value, stability and cetane number.

Cold flow properties

The cold flow properties of a biodiesel are determined by measuring the cold filter plugging point (CFPP) according to test method EN 116. The CFPP indicates the temperature at which saturates can flocculate and viscosity increases. It defines the temperature at which a fuel is no longer filterable within a specified time limit.

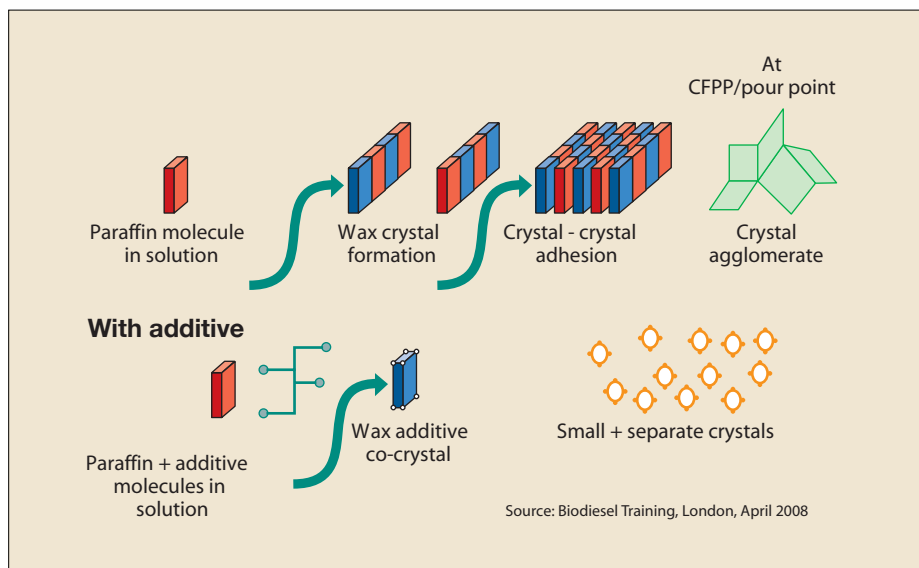


Figure 1 CFPP additives: how do they work?

The CFPP is determined by the original feedstock because it is a function of fatty acid concentration.

For example, PME biodiesel will have a CFPP of around +10°C, in strong contrast to the CFPP of a RME biodiesel of around -10°C. Self-evidently these varieties in cold behaviour properties can be critical for a fuel, depending on the climate and seasonal conditions of the region in which the fuel will be used in a vehicle.

Optimising CFPP can be accomplished by blending biodiesels from several origins with a variety of cold flow properties, or by adding CFPP improvers. CFPP improvers are available commercially, but their effect depends very much on the origins of the biodiesels in which they are used. For example, an additive that works well for RME does not have the same effect for a biodiesel that was derived from PME. If multiple feedstocks are processed for a biodiesel blend, the improver needs to be fully effective for all of the biodiesel types, and this is hard to accomplish. CFPP improvers that are on

the market today work well for a single-source biodiesel, but the manufacturers of these additives are still looking for suitable formulations to cover a blend of biodiesels originating from multiple feedstocks.

Testing CFPP on a laboratory scale is key to the dosage requirements of additives for biofuel blends. A fuels formulator may need to select several types of CFPP additive and test them out on the specific blend ratio of biodiesel that will eventually be blended on a commercial scale at the terminal. Compared to the dosages applied in the mineral diesel industry, higher concentrations of CFPP improvers are needed for biodiesels, depending on the type of additive and the origin of the feedstock.

Another concern is that EN 116 was originally developed for determining the CFPP of mineral diesel with a reproducibility of 3°C. Biodiesel is a biodegradable product, constantly changing, so we may expect that the range of reproducibility for this method is bigger for biodiesel compared to regular diesel fuels. Figure 1 shows how CFPP

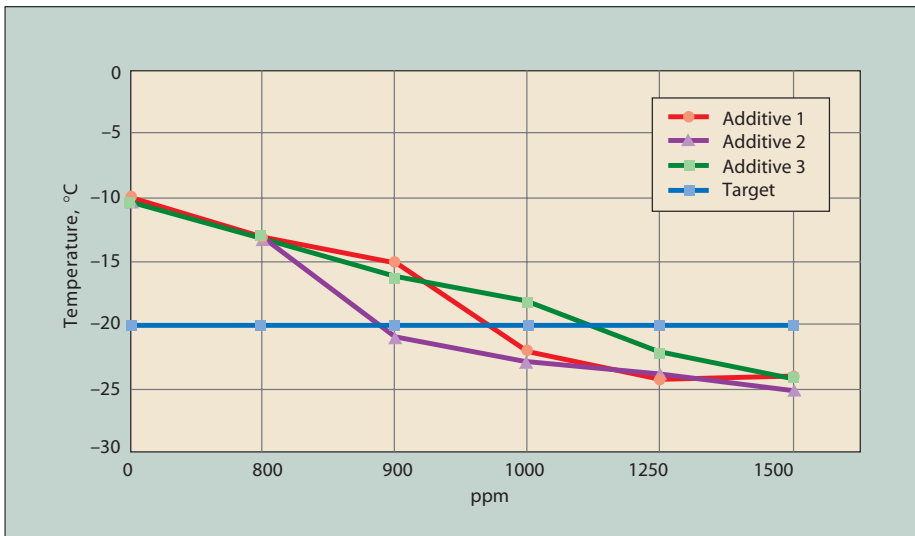


Figure 2 CFPP additive efficiency

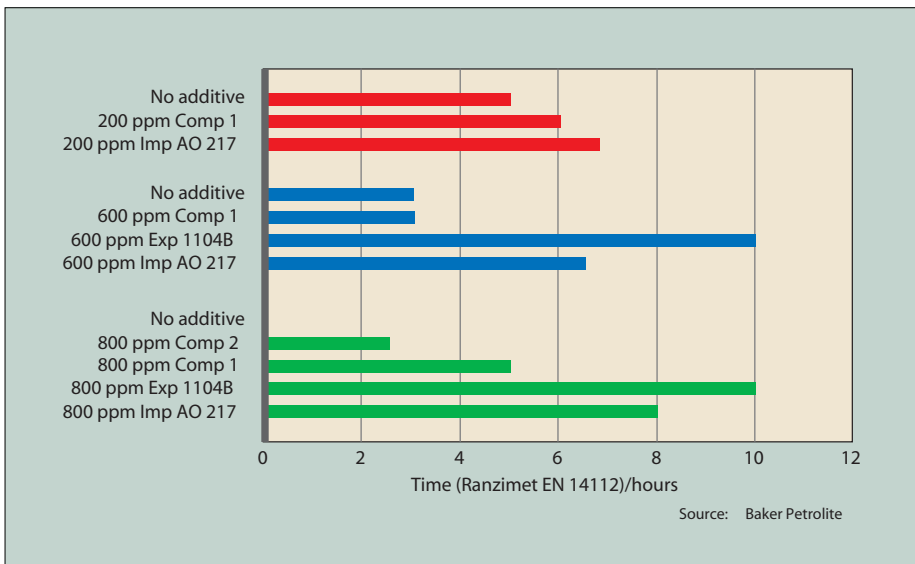


Figure 3 Oxidation stability of biodiesel

additives work and Figure 2 shows the effects of several CFPP additives on a single-source biodiesel (RME).

Iodine value

The iodine value is an indicator of unsaturation in an organic compound and is specific to any type of biodiesel feedstock. It is also an indicator of stability, whereby a high iodine value is related to the level of polymerisation, which may lead to injector fouling. Biodiesels with low iodine values, and therefore relatively low levels of unsaturation, are less sensitive to oxidation processes. European rules specify a maximum 120gm iodine/100gm sample for the EN 14214 iodine specification. This is considered a limitation on the applied concentration ratio of feedstocks with higher iodine values, for instance palm oil.

In trading practice, biodiesels with higher iodine values (mostly derived from the cheapest feedstocks) are blended with biodiesels from other origins with lower iodine values to meet a borderline specification of 120. This specification can be calculated from the iodine values of individual biodiesels to determine the blend ratio.

The reproducibility of this parameter is 3gm iodine/100gm. Hence, the usual recommendation is to calculate the iodine value to 117 and so apply a safety margin, because different laboratories may establish iodine values between 117 and 120 for the same sample.

Stability issues

Since biodiesel is a biodegradable product, it is less stable when compared to mineral

fuels. Depending on the feedstock's origin, the shelf life of biodiesel is limited to between three and six months. This range can be even lower, depending on the exposure to oxidation processes during storage, contact with reactive metal compounds, high water content in the biodiesel, or the type and level of bacteria in contact with the fuel.

Oxidation processes lead to the formation of free fatty acids, which increase the acidity of a biodiesel and eventually form sediments that can block fuel filters.

Commercial additives based on antioxidants such as phenol or amine compounds are available on the market to increase the stability of biodiesels. Recommended application rates are between 100 ppm and 1000 ppm, chiefly depending on the origin of the feedstocks.

At present, the stability specification within EN 14214 requires a minimum of six test hours, which correlates to a six-month period in bulk storage. Discussions are going on within CEN committees to extend this specification to a minimum eight hours, in response to the increase in the variety of feedstocks imported into the EU, which may in turn mean more unstable biodiesels. An example of stability additive performance is shown in Figure 3.

Cetane number

The cetane number is an indicator of a fuel's ignition characteristics in compressed ignition engines, and again the feedstock determines the cetane number of the final product. The minimum requirement for cetane is one of the differentiators between the ASTM & EN biodiesel specifications. The EN specification requires a minimum of 51 compared to a value of 46 in the US.

A cetane index cannot be predicted for biodiesels by calculation, so the cetane number must be measured with a real cetane engine. Alternatively, although this is still not an approved test within EN 14214, you can apply the ignition quality tester (IQT) system, which has some advantages, including faster generation of results and a smaller sample requirement.

Cetane improvers are available to boost the cetane index. These are in the main based on nitrate components. The reproducibility of the test method EN 5165 is to four digits, and even this value is based on round robin results from tests with mineral diesel samples. We can estimate that the reproducibility of the cetane index from a biodiesel sample is even higher compared

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to a petroleum-sourced diesel sample. An example of cetane additive performance is shown in Figure 4.

Sterol glucosides

Over the past 12 months, there have been a few reports of unexpected filter clogging involving diesel fuels blended with biodiesel, even though the biodiesel's cold flow properties did not indicate the likelihood of such problems.

A relationship has been established between filtration and the presence of sterol glucosides. Tests were performed with a sensitive procedure called the filter blocking test specified in the IP 387 test method. These showed that as the level of sterol glucosides increases, so filter blocking tendency (FBT) values increase.

The presence of sterol glucosides at double-digit ppm levels may lead to the formation of a cloudy haze in biodiesel,

even at room temperatures. This haze is the result of agglomerations, between 5 and 15 microns in diameter, of sterol glucosides particles. These agglomerates may also settle at the bottom of a storage tank and hide filter clogging potential as long as they sit undisturbed and undetected.

Yet again, the presence of sterol glucosides depends on the origins of feedstocks used to produce biodiesels. For example, the sterol glucosides content in SME and PME is higher when compared to other harvested oils.

Summary

Biodiesel is a much more sensitive and hygroscopic product than equivalent mineral fuels. Storage and transport guidelines require nitrogen blankets to lower humidity levels and prevent oxidative processes occurring. If biodiesels are selected with poor cold flow properties, there may be a need for heating capacity in shore tanks and vessels to avoid blockages caused by the product during cold weather.

With an increase in the variety of original feedstocks, guaranteeing the quality of product biodiesel has become a complex challenge. Rigorous quality testing and inspection throughout the process, from raw materials testing through processing, transport, storage and blending activities, are key to securing the quality of the fuel for the end consumer.

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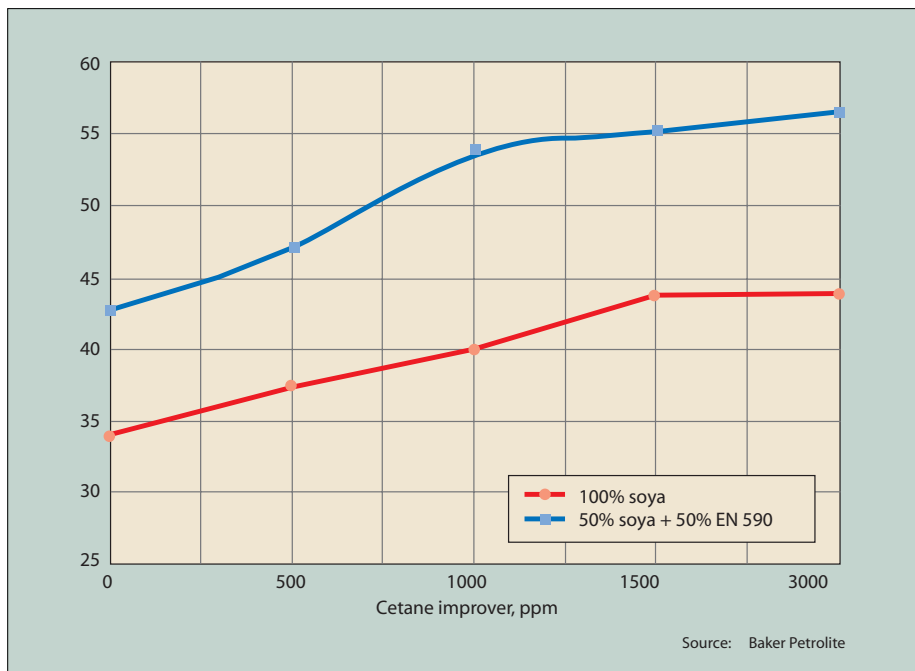


Figure 4 Cetane additive efficiency

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