SHIMADZU

Application Notes

Fourier Transform Infrared Spectrometry

Analysis of Biodiesel Blends by FTIR

Introduction

Biodiesel refers to a diesel-equivalent processed fuel derived from biological sources such as vegetable oils and animals fats. It is also referred to as fatty acid methyl esters (FAME) made from transesterification of vegetable oils or animals fats. Biodiesel contains no petroleum but it can be blended at any level with petroleum diesel to create a biodiesel blend. When biodiesel is blended with petroleum diesel, it produces a fuel that is compatible with diesel engines. Pure biodiesel has high lubricity, high cetane, and a high flash point. "Low blend" can be defined as blends of 5% biodiesel and below. Low blends of biodiesel are also highly effective at enhancing the lubricity of diesel fuel. The typical blend used for lubricity enhancement is 2% biodiesel mixed with 98% diesel (B2).

Fourier transform infrared spectroscopy (FTIR) with suitable accessories can be used effectively to provide quick and non-destructive measurement of FAME in biodiesel blends even for low percentage of blends.

Instrumentation and Experiment

The FAME calibration standards used in the experiment is prepared in accordance to EN standard.

Here Shimadzu FTIR, IRPrestige-21, together with fixed thickness cell and horizontal attenuated total reflection (HATR) attachment were used to generate calibration curves for FAME.

Fixed thickness cell is usually used for quantitative analysis of liquid samples by transmission. Sample is injected into the cell and measurement is taken. For HATR, sample is simply put onto the crystal and covered with a volatile cover for measurement. After the analysis, the crystal is easily cleaned with an appropriate solvent.

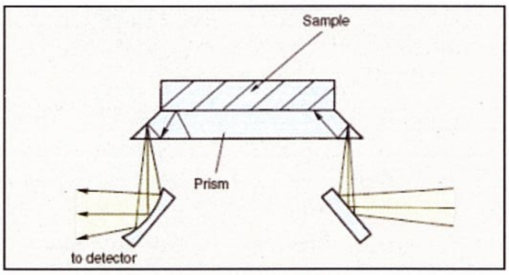

Figure 1. Principle of ATR method

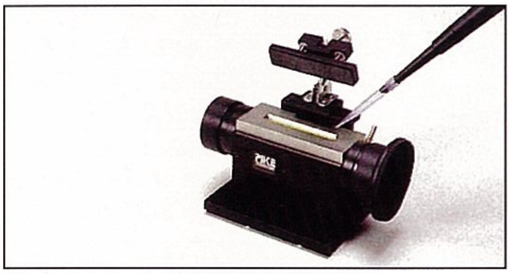

Figure 2. Horizontal ATR Attachment

Results

Figure 3 shows that FAME (from palm olein) has a distinctive sharp carbonyl ester peak at around 1750cm^{-1} which is absent in petroleum diesel. The strong ester peak is thus selected for the calibration of FAME.

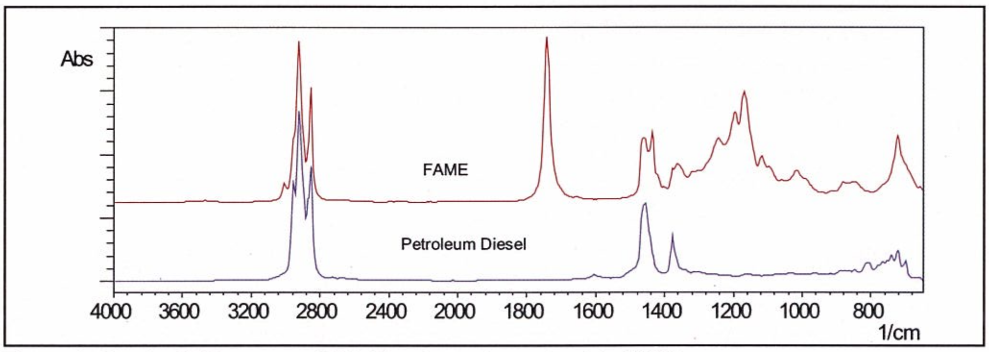

Figure 3. Overlay FTIR spectra of FAME and petroleum diesel by HATR

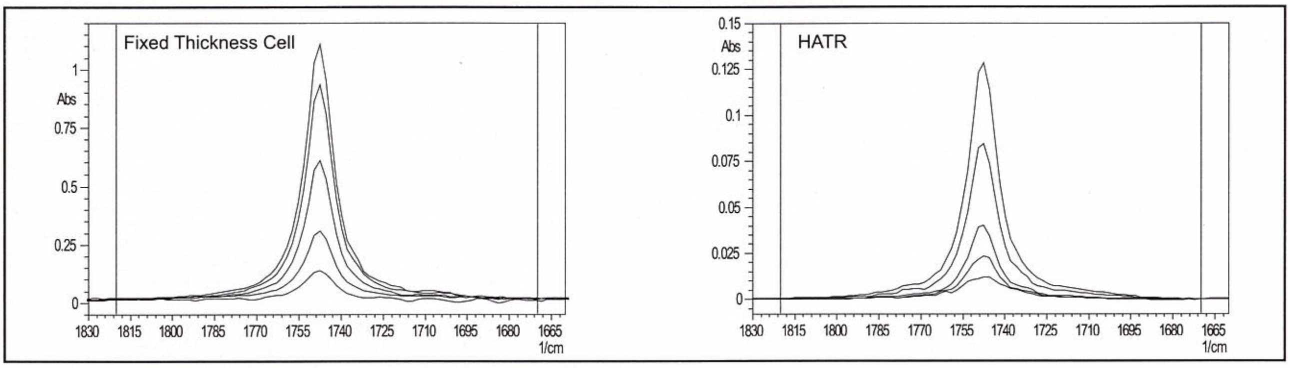

Figure 4. Peak region selected for calibration of FAME

Standard Sample	Peak height Fixed Thickness Cell (NaCl 0.5mm)
0.1% FAME	0.125
0.2% FAME	0.290
0.4% FAME	0.593
0.6% FAME	0.919
0.7% FAME	1.091

Table 1. Peak height of ester peak measured by fixed thickness cell for each FAME standards

Standard Sample	Peak height HATR (ZnSe)
0.4% FAME	0.012
0.6% FAME	0.023
1.0% FAME	0.040
2.0% FAME	0.084
3.0% FAME	0.128

Table 2. Peak height of ester peak measured by HATR for each FAME standards

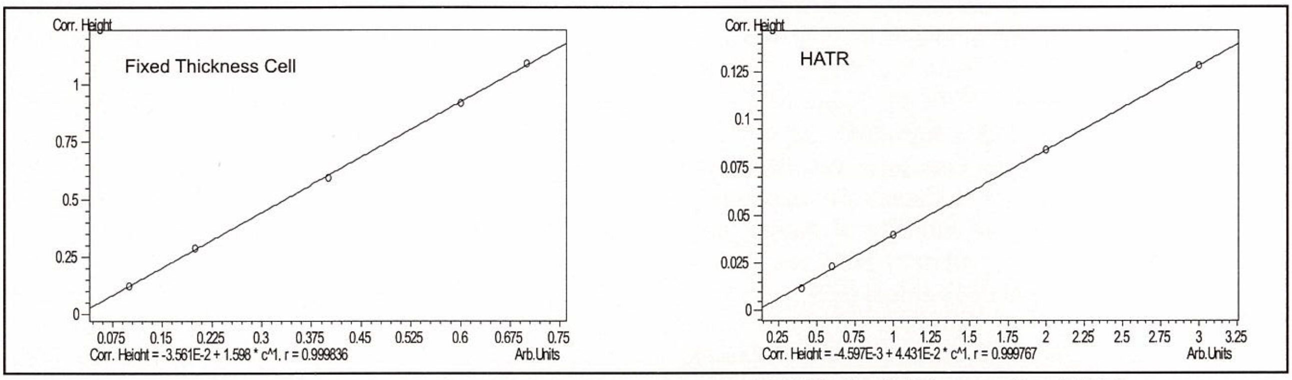

Figure 5. Calibration curves of peak height versus percentage FAME by fixed thickness cell and HATR

Peak height of the ester peak was used for the calibration as shown in Figure 4. Table 1 and 2 show the peak height values for each percentage of FAME measured by fixed thickness cell and HATR. A linear calibration curve of peak height versus concentration was generated as shown in Figure 5. Here, good linearity with $R^2 > 0.9997$ was obtained for both methods.

Summary

For transmission measurement, absorbance intensity is directly proportional to concentration and cell pathlength (Beer-Lambert Law). Fixed thickness cell can be used for the quantitative analysis of low concentration of biodiesel. At high concentration, peak becomes saturated, and dilution is required for absorbance to stay within the linear working range for quantitative analysis. Sensitivity can also be adjusted by selecting different thickness of spacer.

Horizontal attenuated total reflection is a reflection technique. Analysis is usually non-destructive, and is quick and simple as compared to transmission measurement by fixed thickness cell. Peak intensity obtained is lower than that of transmission measurement and higher percentage of biodiesel in blends can be analysed with no peak saturation, hence there is no need for dilution. HATR is sufficient to generate calibration curve for quantitation of biodiesel blends above 0.4%.

Reference

1. http://www.eere.energy.gov/cleancities/blends/biodiesel.html
2. http://www.biodiesel.org/

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