Application Note

050-QU-0220

Quantitative Analysis of Biodiesel (FAME) in Diesel Fuel by FT/IR

1. Introduction

In recent years, Biodiesel fuel made from vegetable oils or cooking oil waste by a transesterification process has been receiving much attention as an alternative energy to processed fossil fuels. The chemical process of transesterification from vegetable oil to FAME is shown in Figure 1.

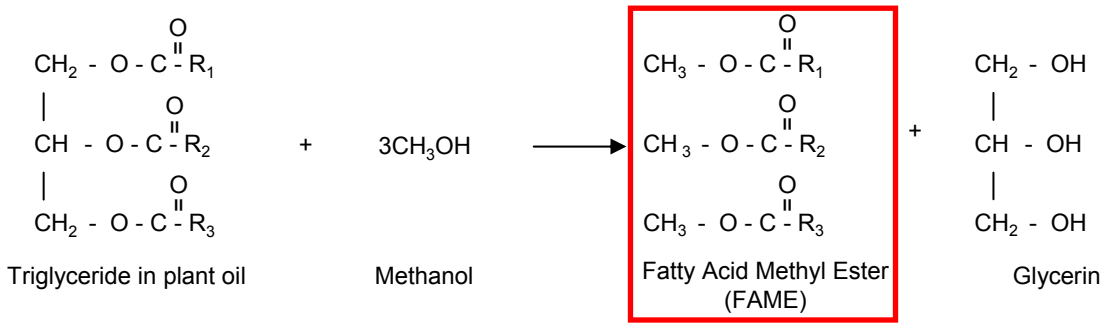

Fig. 1: Reaction pathway from vegetable oil to FAME

The ASTM International and the EN European standards stipulate the determination of fatty acid methyl ester (FAME) in diesel fuel oil and many countries use either neat or blended FAME as biodiesel fuels in accordance with these standards. Since the guidelines differ from country to country for the ratio of FAME blended in diesel oil and other conditions, a simple quantitative analysis method for the determination of FAME in diesel fuel is imperative. The test method of FAME by FT/IR for both the ASTM and the EN standard are shown in Table 1. The ASTM standard requires the ATR method and the EN test method specifies a transmission method. FAME derived from soybean oil used in European industry was used as a sample and a calibration curve model compliant with each standard method was derived. The measurement results obtained are outlined below.

Table 1. Standard Test Methods of FAME by FT/IR

Regulation	ASTM (D7371-07)	EN14078
Measurement method	ATR method	Transmittance using liquid cell
Wavenumber range	4000-650 cm^{-1}	4000-400 cm^{-1}
Software	PLS Quantitative Program	Quantitative analysis program
Instruments	JASCO FT/IR-4100 FT/IR Spectrometer	
Resolution	4 cm^{-1}	
Other measurement conditions	Accumulation: 32 times Accessory: ATR PRO450-S (ZnSe)	Accumulation: 32 times Accessory: Sealed Liquid cell Window material: CaF_2 Pathlength: 0.5 mm

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2. Quantitative Analysis of FAME for ASTM

Figure 2 outlines the infrared spectra of FAME and diesel fuel measured with the ATR method. The PLS calibration curve derived from the standards are shown in Figure 3. The PLS calibration curve was constructed according to the conditions outlined by ASTM using standards that contained the FAME component in the range of 1-10% and using the ATR method. The results demonstrate that PLS and the ATR method is a good combination, which makes reliable results possible without diluting the sample.

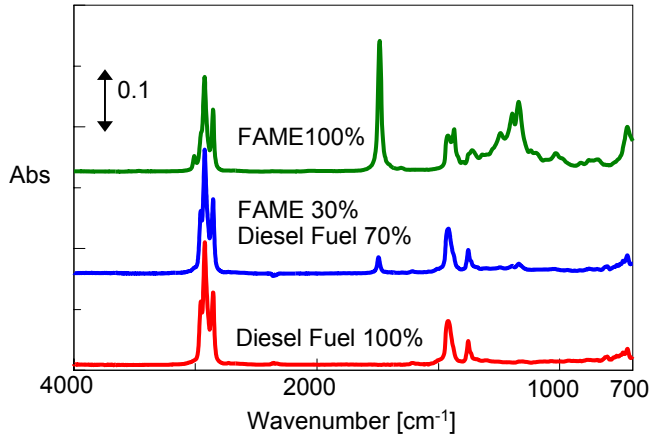

Fig. 2. IR spectra of FAME and diesel oil
(ATR method, resolution: 4 cm^{-1}, accumulations: 32 times)

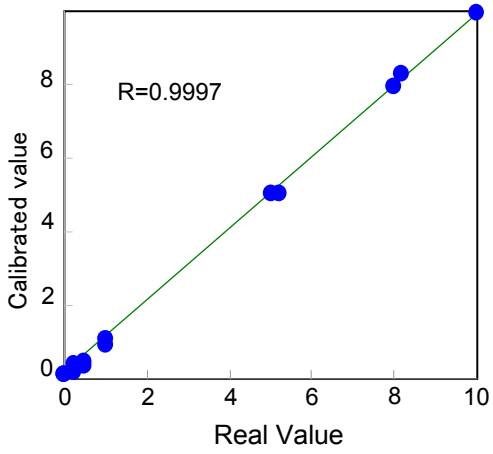

Fig. 3. PLS calibration curve of FAME
Calculation range: 1800-1692, 1327-940 cm^{-1}, No. of Factors: 3

3. Quantitative Analysis of FAME for EN

Figure 4 shows the overlaid peaks for FAME around the 1750 cm^{-1} band from the spectra of the standards obtained by the transmittance method. The standard samples were prepared by mixing 0 - 10% FAME with diesel oil and then diluted 10X in cyclohexane. The calibration curve for the FAME concentration was made by measuring the peak at 1750 cm^{-1} with the standard Quantitative Analysis program as outlined in Figure 5. The results indicate that a linear calibration model was created for the EN standard although the method for the EN standard requires dilution of the sample in cyclohexane.

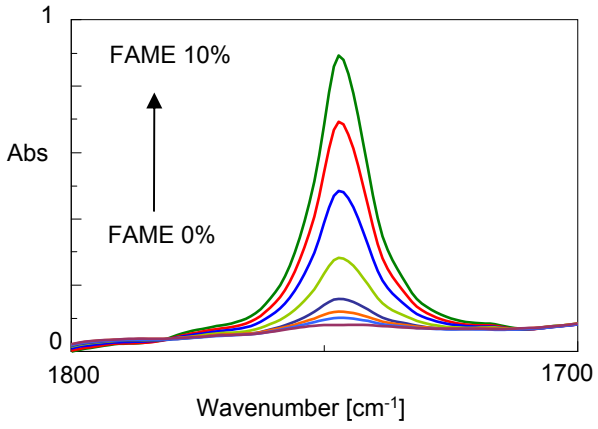

Fig. 4. Peaks of FAME
(Transmission, resolution: 4 cm^{-1}, Accumulation: 32 times)

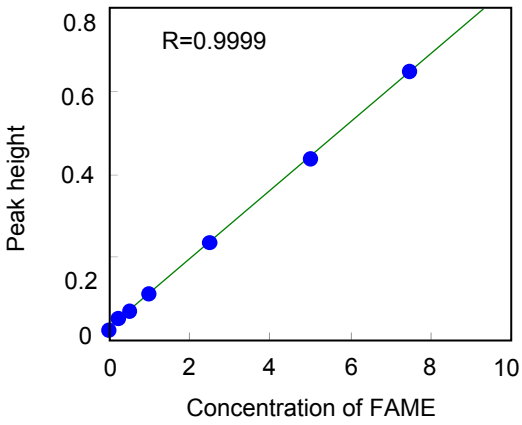

Fig. 5. EN calibration curve of FAME

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