SHIMADZU

Application Notes

Gas Chromatography

Determination of FAME Content in Palm Oil Biodiesel by Gas Chromatography

Introduction

Gas chromatography (GC) is one technique that can be used to test three of the specifications of a pure biodiesel, namely: 1) the FAME contents; 2) the total gycerol contents; and 3) the methanol content.

EN 14214 specifies the minimum FAME content in a pure biodiesel to be 96.5% *m/m*, analyzed by using the standard test method described in EN 14103. The EN 14103 method is suitable for FAME which contains methyl esters of fatty acids having 14 carbon chain (C_{14}) and 24-carbon chain (C_{24}). The method allows one to verify that the ester content of the FAME (biodiesel) is above 90%.

The EN 14103 method provides two ways of calculating the percentage of FAME present in the sample. If the sample is derived purely from vegetable oils, the results based on relative areas is considered to represent percentage by mass. If the sample is derived from non-vegetable oils/fats, the percentage of FAME is determined by using methyl heptadecanoate as an internal standard compound and the following formula:

$$C = [(\Sigma A) - A_{EI}]/A_{EI} \times [(C_{EI} \times V_{EI})/m] \times 100\% \quad (1)$$

where,
ΣA is the total peak area for the methyl esters from C_{14} to $C_{24:1}$;
A_{EI} is the peak area of methyl heptadecanoate;
C_{EI} is the concentration of methyl heptadecanoate solution used;
V_{EI} is the volume of methyl heptadecanoate solution used;
m is the mass of the sample.

Table 1. GC configuration for determination of FAME content in biodiesel.

Gas chromatograph	GC-2010AF
Auto-sampler	AOC-20i
Injector	Split/splitless
Column	FAMEWAX (Restek Corp.), 30 m x 0.25mm, 0.25μm df
Detector	Flame Ionization Detector (FID)
Software	GCsolution

Here we describe an analysis of FAME content in Palm Oil biodiesel by GC-2010A.

Experimental

The system configuration used for this analysis is shown in **Table 1**. Palm oil biodiesel was prepared in our laboratory. 100mg (±10mg) of the palm oil biodiesel sample was weighed into a 10mL vial, then 2.00mL of methyl heptadecanoate solution (10mg/mL) was added to the sample. Then 1μL aliquot of this sample was injected into the GC. The injector was kept at 250ºC, and the injection mode was set to the Split mode (with a split ratio of 50:1). The column temperature was set initially at 200ºC, held for 10min, was subsequently increased to 250ºC at a rate of 20ºC/min, and finally held at 250ºC for 10min. The flame ionization detector was kept at 250ºC during the analysis.

GC-2010AF Gas Chromatography for BioDiesel Analysis.

Results

Although the sample is derived purely from vegetable oils, we used formula (1) to calculate the percentage of FAME in the sample because we believe this will give more accurate results. We determined the FAME content of two aliquots (replicates) of the same biodiesel sample. The FAME content in this biodiesel sample was determined to be 79.1% (see **Table 2**).

Excellent repeatability of injection was obtained for FAME standard compounds, with an average RSD of 0.7% for peak area, and 0.006% for retention time (see **Tables 3** and **4**).

A representative chromatogram of a palm oil biodiesel sample is shown in **Figure 1**. Identification of peaks that are due to fatty acid methyl esters in the sample was based on comparison of the GC-FID data with GC/MS data *(CSC 999 GC-07-10 "Determination of Total Glycerol Content in a Palm Olein Biodiesel by Gas Chromatography", Shimadzu (Asia Pacific) Pte. Ltd., 2007.)*.

Conclusion

Determination of FAME content in biodiesel can be performed in a relatively straightforward procedure with excellent repeatability by using GC-FID.

Table 2. Summary of results of determination of FAME content in a palm oil biodiesel.

Run #	FAME Content in Replicate 1	FAME Content in Replicate 2
1	78.7	79.4
2	78.7	79.4
3	79.1	79.4
Average	78.8	79.4
%RSD	0.29	0.05
Difference (%)	0.6	
Mean FAME content (%)	79.1	

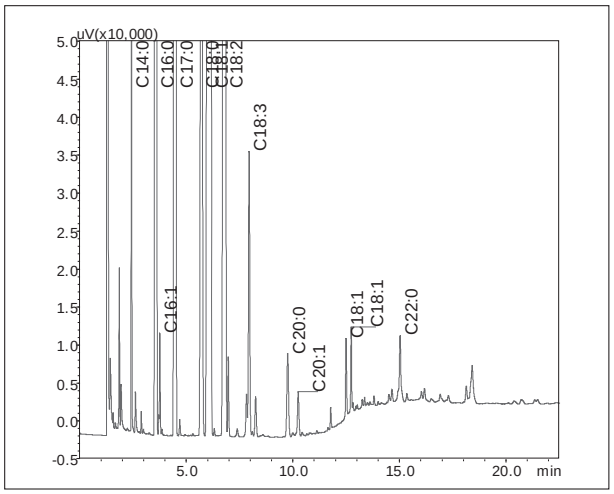

Figure 1. Chromatogram of a palm olein biodiesel fortified with methyl heptadecanoate (C17:0). Proj04_GC8_BioFAME106SPL1.gcd

Table 3. Area repeatability (%RSD) for FAME analysis performed by using GC-2010AF and AOC-20i.

Run #	C14:0	C16:0	C17:0	C18:0	C18:1	C18:2	C18:3
1	280183	274707	209304	267759	292274	275743	171505
2	279817	273901	208394	267309	291416	275224	170680
3	278051	271143	204904	264189	288293	272333	169348
4	277918	271665	205371	265207	289270	273035	169541
5	274206	269360	203358	263716	287591	271625	168820
6	276304	271057	204242	265333	289625	272931	169628
Mean Area	277747	271972	205929	265585	289745	273482	169920
SD	2234	1982	2378	1634	1799	1638	985
%RSD	**0.80**	**0.73**	**1.15**	**0.62**	**0.62**	**0.60**	**0.58**

Table 4. Retention time repeatability (%RSD) for FAME analysis performed by using GC-2010AF and AOC-20i.

Run #	C14:0	C16:0	C17:0	C18:0	C18:1	C18:2	C18:3
1	7.25965	8.39142	8.90020	9.41068	9.51988	9.76963	10.14470
2	7.25948	8.39193	8.90035	9.41100	9.51928	9.76963	10.14532
3	7.25877	8.39065	8.89990	9.40988	9.51880	9.76882	10.14498
4	7.25880	8.39017	8.89915	9.40910	9.51853	9.76803	10.14375
5	7.25875	8.39043	8.90005	9.40960	9.51843	9.76890	10.14413
6	7.25917	8.39105	8.90032	9.41020	9.51902	9.76905	10.14548
Mean RT	7.25910	8.39094	8.89999	9.41008	9.51899	9.76901	10.14473
SD	0.000394	0.000658	0.000447	0.000701	0.000537	0.000597	0.000677
%RSD	**0.005**	**0.008**	**0.005**	**0.007**	**0.006**	**0.006**	**0.007**

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