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Identification of methyl ester content from Waste Cooking Oil Using Gas Chromatographic Method

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Abstract. Waste cooking oil has always been an environment problem in food factories and one method of effect disposing this oil without effecting the environment is to convert it to fatty acid methyl ester (FAME) using small scale pilot plant. The conversion of waste cooking oil with sodium hydroxide as a catalyst in conversional process at 22kHz speed. The reaction of time, molar ratio, speed, catalyst and amount of catalyst will be effect in FAME quality. The quality of biodiesel define is total ester content using gas chromatography. Gas chromatography analysis is a one of technique for identification and quantitation of compounds in a biodiesel sample. From biodiesel sample can identification of contaminants and fatty acid methyl ester. In this research biodiesel sample were analyses using a gas chromatography- flame ionization detector (Perkin Elmer GC Model Clarus 500) equipped with a DB-5 HT capillary column (0.53mm x 5 m) J&W Scientific. The analytic conditions for ester content were as follow by: column temperature used 210^0C, temperature flame ionization detector (FID) of 250^0C, pressure of 80kPa, flow carrier gas of 1ml/min, temperature injector of 250^0C, split flow rate of 50ml/min, time for analysis 20 minute and volume injected of 1 µl. The ester content (C), expresses as a mass fraction in present using formula (EN 14103, 2003a) calculation. Conversion of triglyceride (TG) to FAME using conversional process obtained 96.54 % w.t with methanol to oil molar ratio 6:1, 1%w.t acid sulphuric and 1% w.t sodium hydroxide catalyst.

Introduction

Azhar Food Snd Bhd is a local company producing prawn cracker, snacks and tapioca chips. They used a lot of cooking oil and need to dispose this oil. Waste cooking oil has always been an environment problem in food factories and one method of effect disposing this oil without effecting the environment is to convert it to biodiesel. A pilot plant to produce biodiesel from waste cooking oil (WCO) was designed and installed at the food factory. In this study the waste cooking oil from Azhar Food Snd Bhd was processes to biodiesel where the methyl ester content are calculated follow by European normalization(EN14103) biodiesel standard using gas chromatography flame ionization detector. The quality of biodiesel define is total ester content using gas chromatography .The important of gas chromatography as an analytical tool for organic compounds result from a combination of the separating ability of modern capillary columns, which remains superior to that of pressure- driven liquid separation, and the range of detectors that can be brought to bear to aid solute identification. These detectors, however selective they may be, provide only two-dimensional information- a plot of detector signal versus time. In detector for gas chromatography that provide three dimension of data. These additional data may be used either on their own, or in combination with other information, such as retention time or the output from additional detector, to identify unknown compounds. These are the mass spectrometer, which provides data relating to compound structure: the infrared detector, the output from which is a consequence of chemical functionality, and the atomic emission detector which given information concerning elemental composite.

Methodology

The quality of biodiesel define is total ester content. Limits have been established by the American society for testing and material (ASTM) and the European normalization (EN). The define the minimum to be 96.5 % w.t for fatty acid methyl ester. This is the most important component of biodiesel. The amount of ester in the final product is affected mainly by the extent of tranesterification reaction.

In this research biodiesel sample were analyses using a gas chromatography (Perkin Elmer GC Model Clarus 500) equipped with a DB-5 HT capillary column (0.53mm x 5 m) J&W Scientific. The analytic conditions for ester content were as follow by : coloum temperature used 210^0C, temperature flame ionization detector (FID) of 250^0C, pressure of 80kPa, flow carrier gas of 1ml/min, temperature injector of 250^0C, split flow rate of 50ml/min, time for analysis 20 minute and volume injected of 1 µl. the ester content (C), expresses as a mass fraction in percent, is calculate using following formula (EN 14103, 2003a):

Formula for determine of methyl ester content:

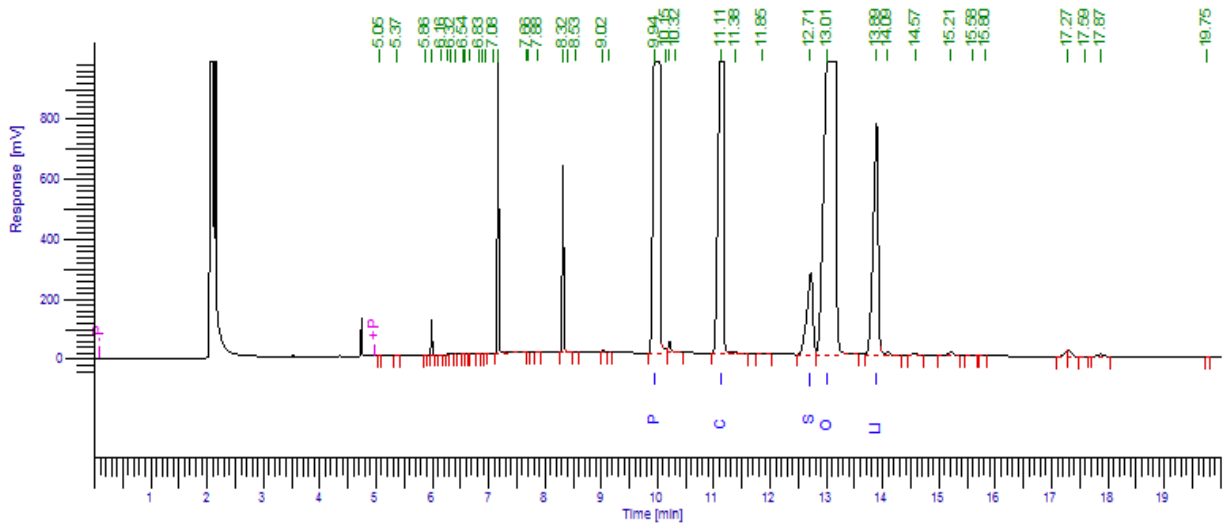
$$C = \frac{(\Sigma A) - A_{EI}}{A_{EI}} \times \frac{C_{EI} \times V_{EI}}{m} \times 100\ \%$$

Where,

ΣA = is the total peak from the methyl ester in C_{14}-C_{24-1}
A_{EI} = is the peak area corresponding to methyl heptadecanoate
C_{EI} = is the concentration, in mg/ml of methyl heptadecanote solution
V_{EI} = is the volume, in ml, of methyl heptadecanote solution
M = is the mass, in mg of the sample

Result and discussion

Determination of methyl ester content referred to EN14105 using gas chromatography- flame ionization detector (FID)



Peak #	Component Name	Time [min]	Area [uV*sec]	Height [uV]
1		5.053	723.60	483.66
2		5.372	1652.37	1064.95
3		5.863	280.21	196.66
4		5.985	144300.28	112505.86
5		6.163	362.16	142.73
6		6.273	1202.56	823.38
7		6.317	1375.71	853.80
8		6.401	2089.91	1752.81
9		6.544	2514.58	2090.42
10		6.581	1586.50	1207.72
11		6.645	315.33	237.16
12		6.831	778.61	362.22
13		6.878	1050.30	538.30
14		6.933	1960.03	1130.40
15		7.084	535.47	140.03
16		7.168	1658941.94	972862.24
17		7.658	10256.10	275.65
18		7.707	2105.08	1548.59
19		7.878	1122.15	439.88
20		8.316	1109323.19	624109.14
21		8.400	8949.18	2490.39
22		8.530	4248.17	1870.80
23		9.025	22327.29	9015.81
24		9.121	2059.61	825.32
25	Palmitic	9.941	8474892.97	972071.92
26		10.149	40432.27	13334.92
27		10.210	127024.34	39082.70
28		10.323	15432.47	3538.58
29	C17	11.115	7041100.89	974232.45
30		11.376	45851.08	5736.00
31		11.845	9864.20	1041.57
32	Stearic	12.712	2064014.94	270712.83
33	Oleic	13.008	14651967.93	977162.81
34	Linoleic	13.887	4717564.08	775400.14
35		14.085	68934.38	10979.68
36		14.570	45840.86	7485.38
37		15.210	95074.79	13523.06
38		15.579	9849.47	1661.22
39		15.805	1094.51	176.66
40		17.270	76004.48	20720.33
41		17.289	99922.40	20793.03
42		17.594	5474.04	896.64
43		17.873	75792.97	8684.78
44		19.751	2583.53	1598.54
			40648776.96	5.86e+06

Figure 1: Analysis FAME from WCO using gas chromatography- flame ionization detector (FID)

Formula for determine of methyl ester content:

$$C = \frac{(\Sigma A) - A_{EI}}{A_{EI}} \times \frac{C_{EI} \times V_{EI}}{m} \times 100\ \%$$

ΣA (C_{14}-C_{24-1})
= (144300.28 + 1658941.94 + 1109323.19 + 22327.29 + 8474892.97 + 40432.27 + 127024.34 + 15432.47 + 7041101 + 2064015 + 14651968 + 4717564.08 + 68934.38 + 45840.86 + 95074.79 + 76004.48 + 99922.4 + 75792.97) = 41028892.5

A_{EI} = 5028356.15
C_{EI} = 10mg/ml
V_{EI} = 5 ml
M = 250 mg

$$C = \frac{(41028892.5 - 701101)}{701101} \times \frac{10\ mg/ml \times 5ml}{250mg} \times 100\% = 96.54\ \%$$

From the result showed that the molar ratio of WCO to MeOH of 6:1, NaOH catalyst of 1% and agitation speed 20kHz , produced ester content 96.54% wt.

Summary

Gas Chromatography is one of the most important tools in chemistry because of its simplicity, sensitivity and effectiveness in separating components of mixtures. It is widely used for quantitative and qualitative analysis of mixtures and for the purification of compounds. Gas Chromatography can be defined as differential migration processes where gases or vaporized sample components are selectively retained by a stationary phase. Method EN 14103 is used to determine the fatty acid methyl ester (FAME) between C14:0 and C24:1 and linolenic acid methyl ester content of oil feedstock used in biodiesel production. EN 14103 calls for calibration of all FAME components by relative response to a single compound – methyl heptadecanoate. The conversional process obtained conversion waste cooking oil to FAME about 96.54 %w.t with methanol to oil ratio 6:1, 1% acid sulfuric and sodium hydroxide as ctatalyst and 2hour reaction time.

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