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APPENDICES

APPENDIX A

EXPERIMENTAL AND DATA ANALYSIS

A-1 Acid Value (Free Fatty Acid Content), AOCS Official Method Cd-3D-63

1. Weigh FFA 100% 10-20 g. in conical flasks 250 ml.
2. Add mix equal volumes of 96% ethanol and diethyl ether molar ratio 1:1 50 ml.
3. Solution is titrated with 0.5 N sodium hydroxide solution.
4. Titrate while swirling, using phenolphthalein as indicator.

Calculation

Let: Weight (in g.) of sample = w

Volume (in ml.) of sodium hydroxide used = v

Normality of sodium hydroxide = N

Molecular weight of the fatty acids = M

$$\text{Acid value} = \frac{56.1Nv}{w}$$

$$\text{Free fatty acid (FFA) \%} = \frac{vNM}{10w}$$

A-2 Saponification Value, AOCS official Method Cd-3b-76

The saponification value is determined by completely saponifying the oil or fat with a known amount of potassium hydroxide, the excess of which is determined by titration.

Reagents

Hydrochloric acid 0.5 N aqueous solution, accurately standardized.

Potassium hydroxide 0.5 N solution in 96% ethanol.

Phenolphthalein indicator 1% in 96% ethanol.

Apparatus

Conical flasks 250 ml.; made of alkali-resistant glass; provided with a reflux condenser with a ground joint.

Process

1. Weigh into a 250 ml. conical flask about 4 g. filtered fat with an accuracy of 1mg.
2. Add, accurately measured, 50 ml. 0.5 N ethanol potassium hydroxide solution to the cold fat and attach the reflux condenser to the flask.
3. Heat, and as soon as the ethanol boil, occasionally shake the flask until the fat is completely dissolved. Boil the solution for half an hour after the fat is completely dissolved.
4. Add 1 ml. phenolphthalein indicator and slowly titrate the hot soap solution obtained with 0.5 hydrochloric acid.
5. Carry out a blank determination upon the same quantity of potassium hydroxide solution at the same time and under the same conditions.

Calculation

Let; Weight (in g.) of oil or fat taken = w

Volume (in ml.) of hydrochloric acid used in test = v_1

Volume (in ml.) of hydrochloric acid used in blank = v_2

Normality of hydrochloric acid = N

$$\text{Saponification value (S.V.)} = \frac{56.1N(V2-V1)}{w}$$

For the determination of the mean molecular weigh of the fatty acids present in a fat the following methods may be used;

1. Assuming the fat to consist of a mixture of triglycerides and free fatty acids and fixed and free fatty acids to have the same mean molecular weight, an apparent value for the mean molecular weigh of the fatty acids (M) may be calculated:

$$M = \frac{[56108 - 12.67(SV\text{-}AV)]}{SV}$$

Where

SV = saponification value of the fat

AV = acid value of the fat

2. The mean molecular weight of the water-insoluble fatty acids (M) is calculated:

$$M = \frac{56108}{\text{SV of fatty acids}}$$

3. In 1 and 2 the unsaponifiable is included in M. The true mean molecular weight of the water insoluble fatty acids is determined by saponification of the fat, extraction of the unsaponifiable matter, separation of the fatty acids and determination of their saponification value:

$$M = \frac{56108}{\text{SV of fatty acids}}$$

Note: The saponification value (S.V), which is related to the molecular weigh of the fat, denotes the number of mg. potassium hydroxide which is required to

saponify 1 g. of fat, i.e. to neutralize the free fatty acids and the fatty acids combined as glycerides.

A-3 Calculation molecular weight of palm fatty acid

Saponification value of palm fatty acid, AOCS official Method Cd-3b-76

Calculation

Let; Weight (in g.) of oil or fat taken = w

Volume (in ml.) of hydrochloric acid used in test = v_1

Volume (in ml.) of hydrochloric acid used in blank = v_2

Normality of hydrochloric acid = N

$$\text{Saponification value} = \frac{56.1N(V2-V1)}{w}$$

$$\text{S.V.} = \frac{56.1\times0.5\times(22-7.5)}{2}$$

$$= 203.3625$$

For the determination of the mean molecular weigh of the fatty acids present in a fat the following methods may be used;

1. Assuming the fat to consist of a mixture of triglycerides and free fatty acids and fixed and free fatty acids to have the same mean molecular weight, an apparent value for the mean molecular weigh of the fatty acids (M) may be calculated:

Where

SV = saponification value of the fat

AV = acid value of the fat

$$M = \frac{[56108 - 12.67(SV\text{-}AV)]}{SV}$$

$$= \frac{56108 - (12.67)(\text{S.V. neutral})}{\text{S.V. neutral}}$$

$$\text{S.V. neutral} = \frac{56108 \times \text{S.V.}}{56108+12.67(\text{A.V.})}$$

$$= \frac{56108 \times 203.3625}{56108+12.67(147.5)}$$

$$= 196.808$$

$$\text{M.W.} = \frac{56108 - 12.67(196.808)}{196.808}$$

$$\text{M.W.} = 272.42$$

A-4 Calculation molecular weight of palm stearin

Saponification value of palm fatty acid, AOCS official Method Cd-3b-76

Calculation

Let; Weight (in g.) of oil or fat taken = w

Volume (in ml.) of hydrochloric acid used in test = v_1

Volume (in ml.) of hydrochloric acid used in blank = v_2

Normality of hydrochloric acid = N

$$\text{Saponification value (S.V.)} = \frac{56.1N(V2-V1)}{w}$$

$$\text{S.V.} = \frac{56.1*0.5*(22-8.2)}{2}$$

$$= 193.54$$

The mean molecular weigh of the fatty acids (M) may be calculated:

$$\text{M} = \frac{[56108-12.67(SV-AV)}{SV}$$

$$= \frac{56108-(12.67)(193.54-0.31)}{193.545}$$

$$= 277.24$$

$$\text{3M.W.} = 831.72$$

APPENDIX B

CALCULATION OF PERCENT OF METHYL ESTER

B-1 Calibration curve of fatty acids methyl esters.

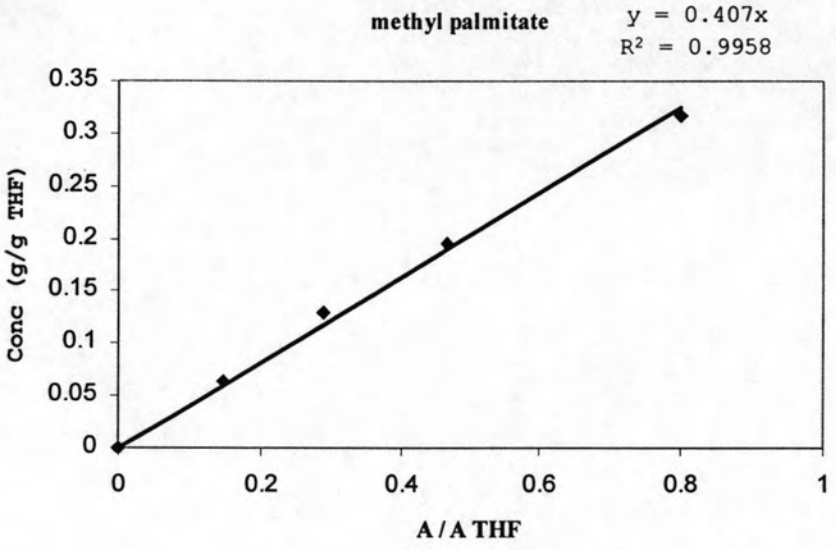


Figure B-1 Calibration curve of methyl palmitate.

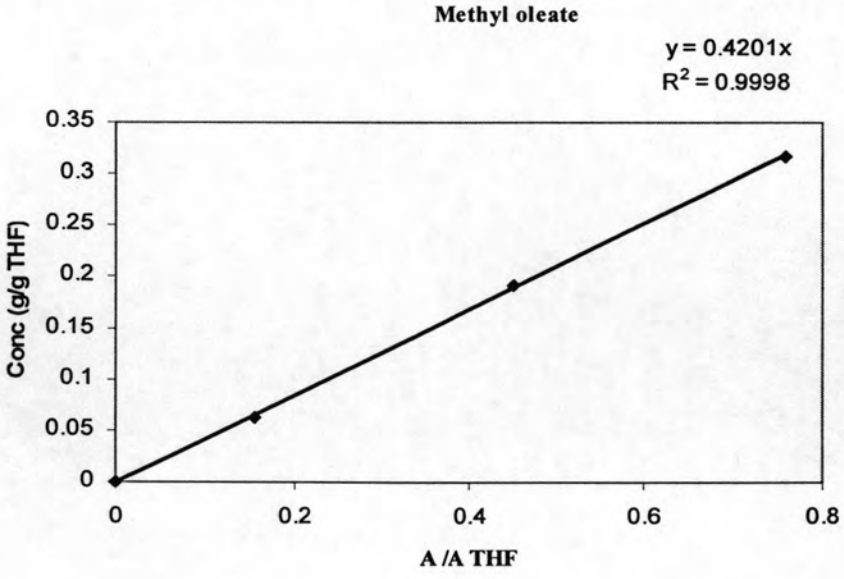


Figure B-2 Calibration curve of methyl Oleate

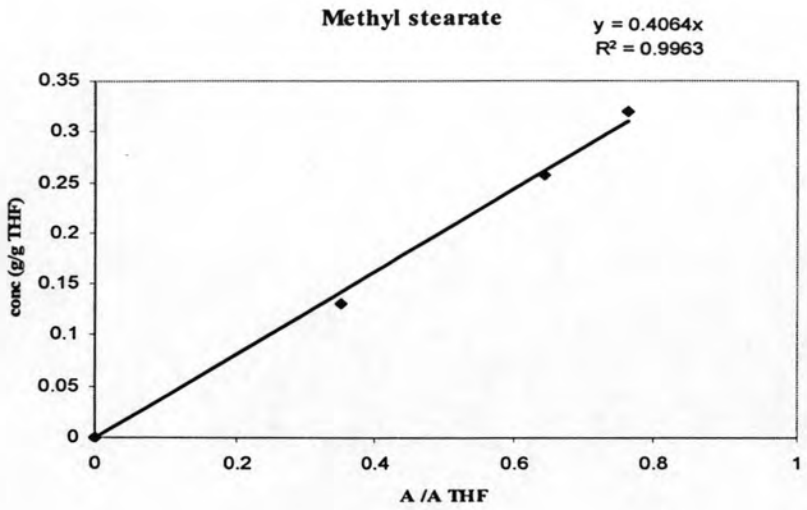


Figure B-3 Calibration curve of methyl Stearate.

B-2 Analysis of methyl ester

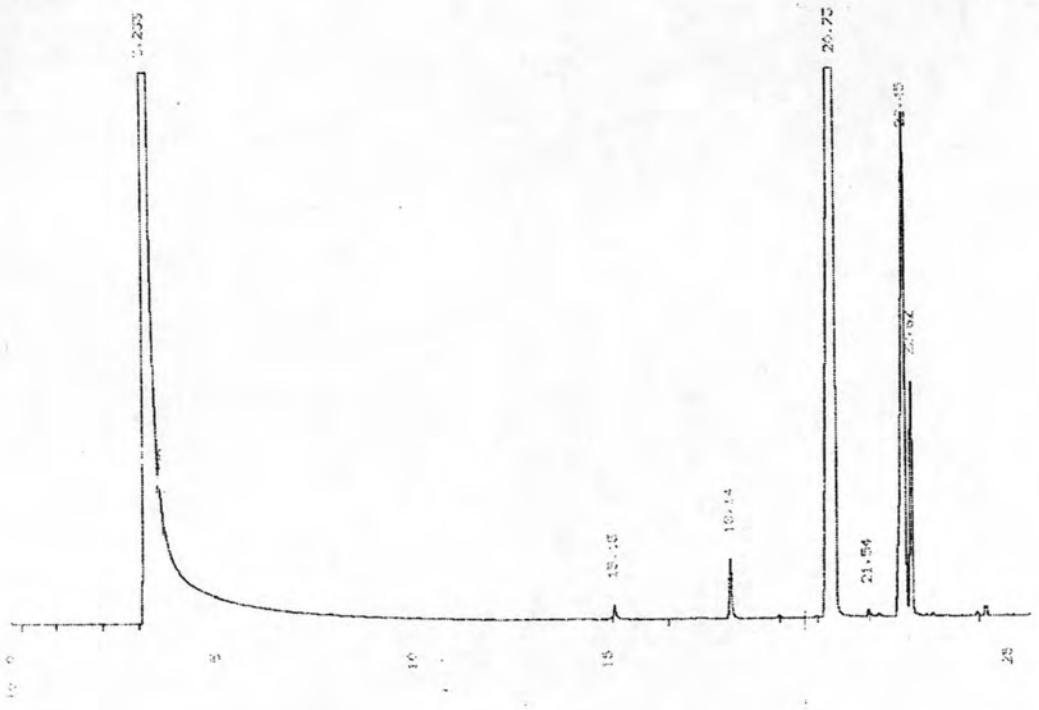
Analysis of methyl ester in product by gas chromatography (GC). The reaction time of each methyl ester is different. Therefore, for find the type of methyl ester by compare retention time of each methyl ester with methyl ester standard. The retention time are shown in Table B-1.

Table B-1 Retention time of methyl ester in GC chromatogram

Number of peak	Retention time (min)	Peak of sample
1	3.33	Tetrahydrofuran
2	20.76	Methyl palmitate
3	22.69	Methyl oleate + Methyl linoleate
4	22.94	Methyl stearate

B-3 GC chromatogram of methyl ester from experiment

From experiment esterification and transesterification using acid and base catalysts can see main methyl ester

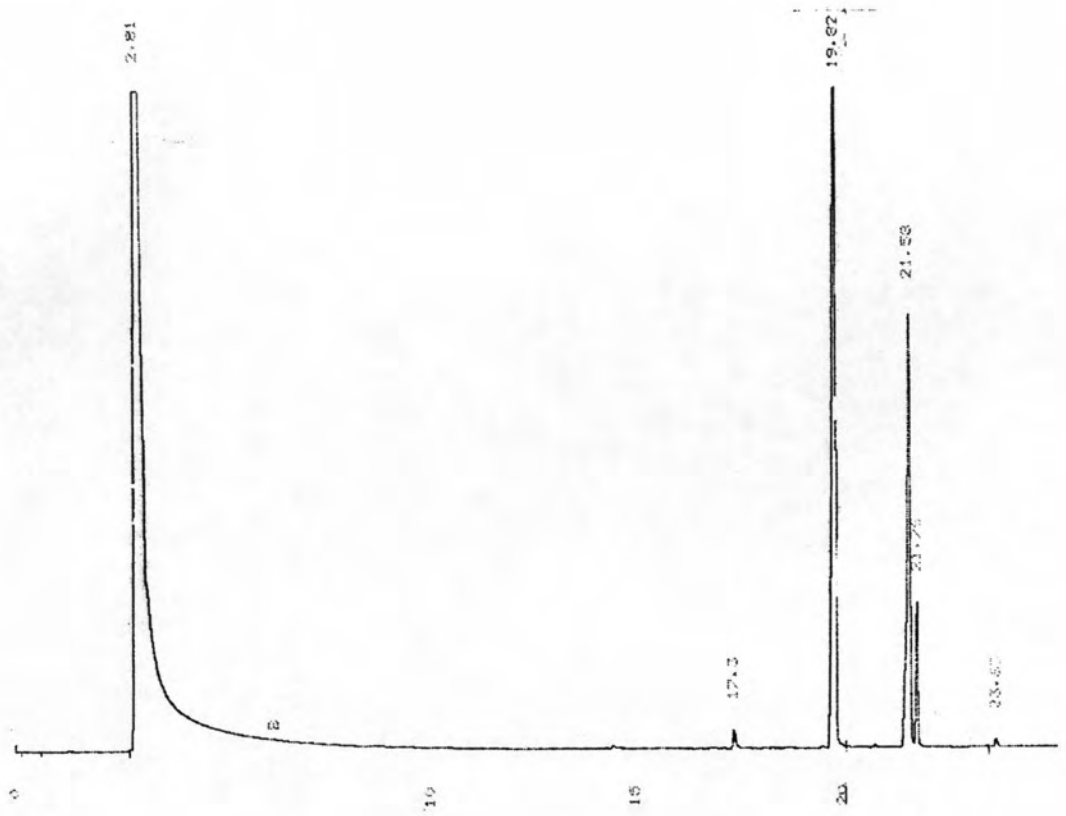


DETECTOR 2

RT	AREA	BC	AREA%
1.643	0.0920	V	0.0537
2.419	0.0559	V	0.0326
3.233	82.4511	T	48.1381
3.720	12.4232		7.2531
15.15	0.3860	V	0.2253
18.14	1.0575	T	0.6174
19.36	0.1099	V	0.0642
20.73	52.0510	T	30.3893
21.54	0.3253	T	0.1899
22.45	16.0834	T	9.3901
22.62	4.6553	T	2.7179
24.25	0.0876	T	0.0511
24.46	0.4319	T	0.2521
25.61	0.2628	T	0.1534
26.26	0.8069		0.4711

15 PEAKS > AREA/HT REJECT

Figure B-4 Chromatogram for methyl ester at condition: Palm stearin reactant, 300%mol excess of methanol from its stoichometric, 1.0% KOH, 60 min and 60°C



DETECTOR 2

RT	AREA	BC	AREA%
2.81	65.9866		68.9876
17.39	0.4884	V	0.5106
19.82	17.8322	T	18.6432
21.58	9.9600	T	7.3675
21.76	1.9331	V	2.0210
23.62	0.3436	V	0.3592
27.15	0.1058	V	0.1106

7 PEAKS > AREA/HT REJECT

Figure B-5 Chromatogram for methyl ester at condition: FFA reactant, 300%mol excess of methanol from its stoichometric, 1.0% H_2SO_4,60min and 60°C.

APPENDIX C

EXPERIMENTAL DATA OF TRANSESTERIFICATION AND ESTERIFICATION USING BASE AND ACID CATALYSTS

Table C-1 The concentration of methyl ester (wt%) from palm stearin at 60°C and 60 min

%mol excess of methanol	%KOH	Concentration of methyl ester (wt%)			
		Palmitate	Oleate+Linoleate	Stearate	Total
100	0.5	31.43	36.78	3.93	72.16
300	0.5	33.92	37.21	6.81	77.94
100	1.0	62.00	19.31	5.60	86.91
300	1.0	67.09	21.36	5.98	94.44

Table C-2 The concentration of methyl ester (wt%) from palm fatty acid at 60°C and 60min

%mol excess of methanol	$\%H_2SO_4$	Concentration of methyl ester (wt%)			
		Palmitate	Oleate+Linoleate	Stearate	Total
100	0.5	23.38	18.67	3.44	45.50
300	0.5	21.34	20.51	4.32	46.19
100	1.0	24.04	21.66	3.58	49.29
300	1.0	35.04	17.11	3.57	55.73

Table C-3 The concentration of methyl ester (wt%) from palm fatty acid to palm stearin use 0.5%H_2SO_4

Fatty acid	%mol excess of methanol	Concentration of methyl ester (wt%)			
		Palmitate	Oleate+Linoleate	Stearate	Total
0%FFA	100	4.10	2.06	0.29	6.45
0%FFA	300	5.06	2.46	1.40	8.91
25%FFA	100	9.28	5.22	2.18	16.68
25%FFA	300	10.08	5.84	1.57	17.49
50%FFA	100	10.88	6.45	0.91	18.23
50%FFA	300	13.46	9.38	2.79	25.63
75%FFA	100	17.23	15.47	2.78	35.48
75%FFA	300	19.61	16.00	2.62	38.23
100%FFA	100	23.38	18.68	3.44	45.50
100%FFA	300	21.35	20.52	4.33	46.20

Table C-4 The concentration of methyl ester (wt%) from palm fatty acid to palm stearin use 1.0%H_2SO_4

Fatty acid	%mol excess of methanol	Concentration of methyl ester (wt%)			
		Palmitate	Oleate+Linoleate	Stearate	Total
0%FFA	100	11.58	5.53	1.38	18.49
0%FFA	300	12.36	7.45	1.55	21.36
25%FFA	100	13.83	9.48	2.45	25.76
25%FFA	300	16.21	11.49	1.46	29.15
50%FFA	100	13.99	11.15	2.73	27.88
50%FFA	300	17.86	13.02	2.56	33.44
75%FFA	100	17.12	16.35	6.39	39.86
75%FFA	300	23.24	19.61	2.95	45.81
100%FFA	100	25.08	20.49	3.14	48.71
100%FFA	300	24.04	21.66	3.58	49.29

Table C-5 The percent of methyl ester from palm fatty acid to palm stearin use 0.5%KOH

Fatty acid	%mol excess of methanol	Concentration of methyl ester (wt%)			
		Palmitate	Oleate+Linoleate	Stearate	Total
0%FFA	100	31.43	36.78	3.93	72.16
0%FFA	300	33.92	37.21	6.81	77.94
25%FFA	100	4.93	5.34	9.63	19.90
25%FFA	300	5.01	10.47	4.49	19.97
50%FFA	100	4.95	7.49	0.95	13.38
50%FFA	300	5.04	4.59	5.88	15.51
75%FFA	100	4.68	3.91	0.76	9.35
75%FFA	300	4.36	5.48	0.65	10.50
100%FFA	100	4.51	2.38	0.50	7.39
100%FFA	300	4.49	2.17	0.95	7.61

Table C-6 The concentration of methyl ester (wt%) from palm fatty acid to palm stearin use 1.0%KOH

Fatty acid	%mol excess of methanol	Concentration of methyl ester (wt%)			
		Palmitate	Oleate+Linoleate	Stearate	Total
0%FFA	100	62.00	19.31	5.60	86.91
0%FFA	300	67.09	21.36	5.98	94.44
25%FFA	100	4.89	11.69	4.39	20.97
25%FFA	300	4.97	13.23	4.89	23.09
50%FFA	100	5.48	5.73	5.53	16.74
50%FFA	300	4.83	3.07	9.75	17.65
75%FFA	100	4.74	3.06	2.67	10.46
75%FFA	300	4.90	7.68	0.58	13.16
100%FFA	100	4.28	4.44	0.31	9.03
100%FFA	300	4.37	3.51	2.52	10.39

VITA

Tulaporn Treesutat was born on 8 October, 1982 in Singburi, Thailand. She received Bachelor's Degree of Chemical Engineering from the Faculty of Engineering Srinakharinwirot University in 2004. After then she subsequently completed the requirements for a Master's Degree in Chemical Engineering at the Department of Chemical Engineering, Faculty of Engineering, Chulalongkorn University in 2006.