CHAPTER IV

RESULTS AND DISCUSSION

Palm oil was used as main raw material through out this research. The ^{13}C-NMR spectrum of palm oil was shown in Figure A1.

Figure A1, ^{13}C-NMR spectrum, showed the important signal of triglyceride, —CH_2—O—, of palm oil at 62.02 and 68.86 ppm and signal of C=O appeared at 173.5 ppm. In addition, Figure A1 also showed signal of unsaturated group at between 126 and 130 ppm.

This research was studied in two ways of the reaction. The first way was consisted of two distinct processes : transesterification and hydrogenation. The second way consistsed of two distinct processes : transesterification and hydroxylation.

Transesterification

Transesterification process was carried out to give monoester from palm oil, which was mainly a triglyceride of oleic acid and palmitic acid, by reacting with an alcohol (1-butanol, 1-hexanol, cyclohexanol, or 2-ethyl-1-hexanol) and using concentrated sulfulic acid as a catalyst.

The optimum condition for transesterification with each alcohol was obtained by varying reaction temperature and reaction time. In this study, the

reaction temperature was varied from 70, 80, and 90 ^{O}C. While the reaction was performed at various temperature, the reaction time was studied between 1 and 3.5 hours.

Characteritics of monoester products were determine by ^{13}C-NMR and GC-MS.

The ^{13}C-NMR spectrum of 1-butanol, 1-hexanol, cyclohexanol, and 2-ethyl-1-hexanol were shown in Figure A2, A3, A4, and A5, respectively.

1. Transesterification of palm oil with 1-butanol

The results of butyl ester from transesterification at optimum condition were shown by ^{13}C-NMR and GC-MS spectrum in Figure A6 and A18 , respectively.

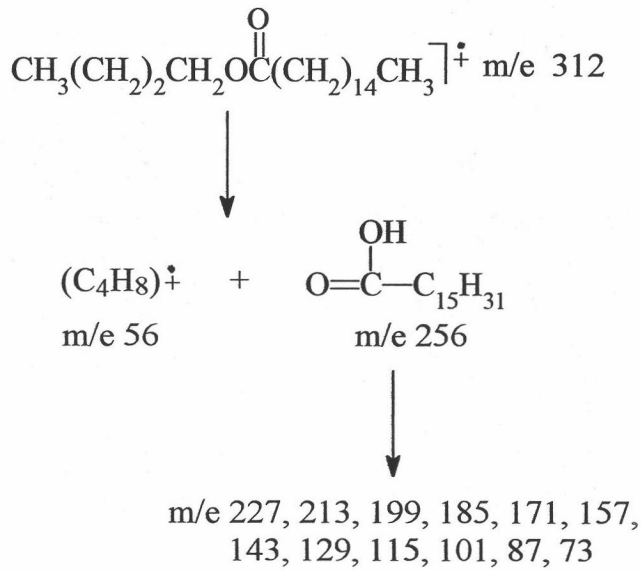
From Figure A6, ^{13}C-NMR spectrum, when reaction temperature was 80 ^{O}C and reaction time was 1 hour, the result demonstrated that the peak of triglyceride of palm oil at 62.02 and 68.86 ppm disappeared and the important peak of —CH_2—O— and C=O of monoester product appeared at 63.8 and 173.5 ppm, recpectively. In addition, Figure A6 also showed peak of unsaturated group at between 126 and 130 ppm.

These experimental results indicated that the transesterification reaction of palm oil with 1-butanol was complete at reaction temprature of 80 ^{O}C and reaction time of 1 hour. In this study, the yield of the resulting product was 92.57%.

The composition of butyl ester product was determined by GC-MS. The GC-MS was performed in a DB-1 capillary column. The GC-MS chromatogram was shown in Figure A18 and Mass spectrum of each peak was shown in Figure A19 to A20.

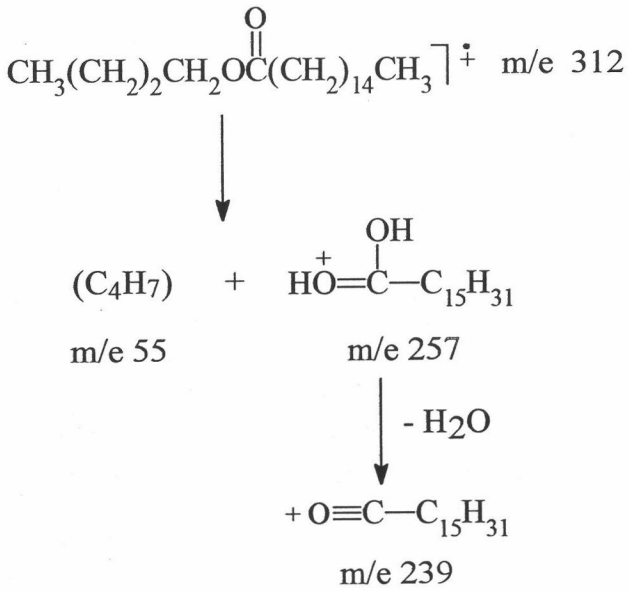
Figure A18, GC-chromatogram, indicated that butyl ester product was composed of a mixture of butyl ester of long chain fatty acid. The main composition were butyl palmiate and butyl oleate, at retention time 8.76 and 10.55, recpectively.

Mass-spectrum of butyl palmitate (MW 312), at retention time 8.76, in Figure A19 showed base peak at 56 due to McLafferty rearrangement as the following equation :



The reaction could occur such as shown in the following equation (I and II) :

- Equation I : double H-shift



- Equation II : McLafferty rearrangement

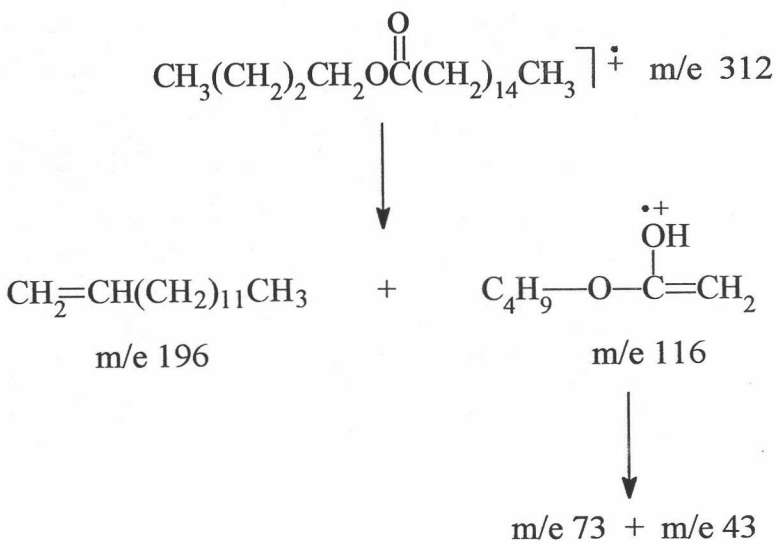
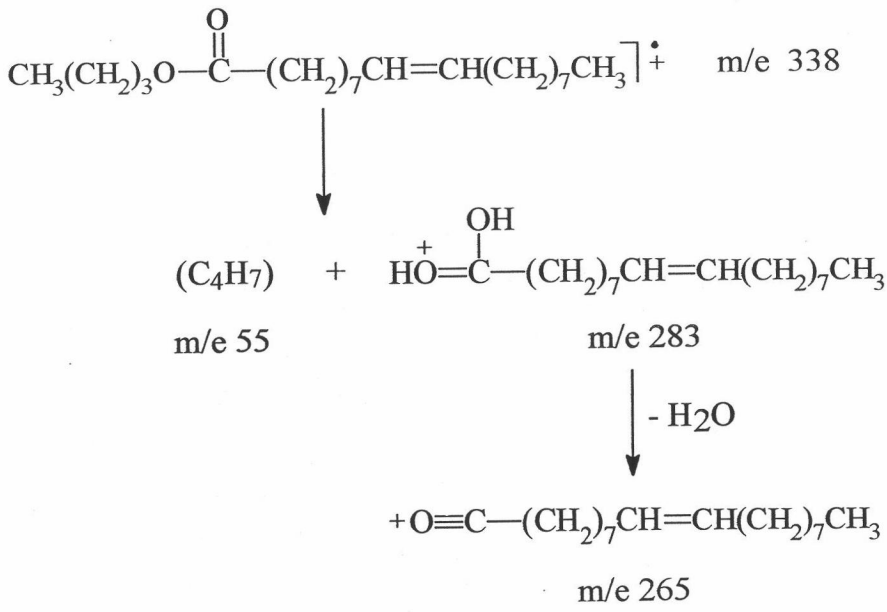
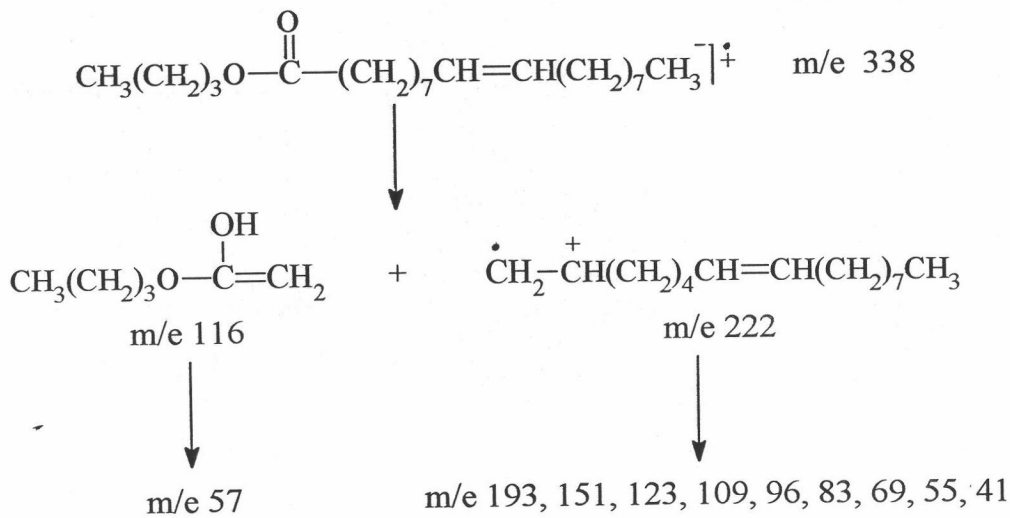


Figure A19 , Mass-spectrum of butyl oleate (MW 338), at retention time 10.55, showed base peak at 55 due to double H-shift as the following equation :



The reaction could occur as shown in the following equation :



The physical and chemical properties of butyl ester product, as showed in Table 4-1, were studied as follow : color, pour point, kinematic viscosity at 40 and 100 ^{O}C, viscosity index, flash point, and oxidation stability. The oxidation and thermal stability curve were analyzed by TGA analyzer and the results were shown in Figure A41.

Table 4-1 : The physical and chemical properties of palm oil and butyl ester product.

Properties	Palm Oil	Butyl Ester Product
Color, ASTM	1.0	L2
Kinematic Viscosity		
@ 40 ^{O}C, cSt	40.26	6.04
@ 100 ^{O}C, cSt	8.37	2.26
Viscosity Index	190.48	197.04
Pour Point, ^{O}C	+12	-6
Flash Point, ^{O}C	314	215
Oxidation Point, ^{O}C	385	365
Oxidation Compounds, %wt	37.37	10.96

The result from Table 4-1 indicated that the pour point of product was -6 ^{O}C. Flash point was 215 ^{O}C. The viscosity at 40 and 100 ^{O}C were 6.04 and 2.26 cSt, respectively, and the viscosity index was 197.04. The color was lower than 2. The oxidative compounds were 10.96 %wt.

2. Transesterification of palm oil with 1-hexanol

The results of hexyl ester from transesterification at optimum condition were shown by as ^{13}C-NMR and GC-MS spectrum in Figure A8 and A24, respectively.

From Figure A8, ^{13}C-NMR spectrum, when the reaction temperature was 80 ^{O}C and the reaction time was 1.5 hours, the result demonstrated that the peaks of triglyceride of palm oil at 62.02 and 68.86 ppm disappeared and the important peaks of —CH_2—O— and C=O of monoester product appeared at 64.1 and 173.5 ppm, recpectively. Furthermore, Figure A8 also showed peak of unsaturated group at between 126 and 130 ppm.

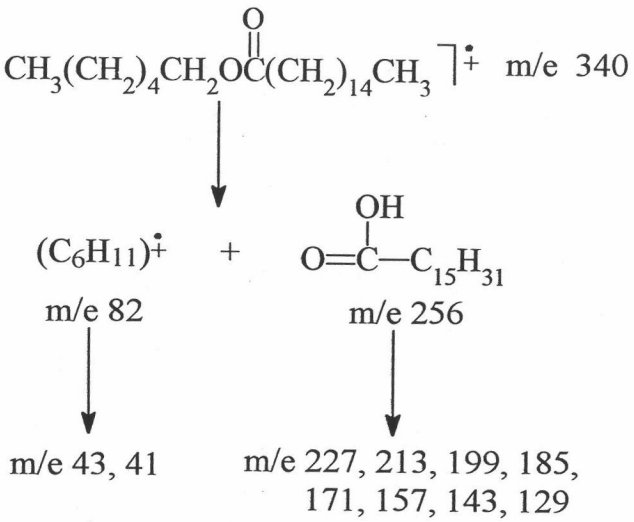
These experimental results indicated that the transesterification reaction of palm oil with 1-hexanol was complete at reaction temprature of 80 ^{O}C and reaction time of 1.5 hours. In this study, the yield of the resulting product was 90.57 %.

The composition of hexyl ester product was determined by GC-MS. The GC-MS was performed in a DB-1 capillary column. The GC-MS chromatogram was shown in Figure A24 and Mass spectrum of each peak was shown in Figure A25 to A26.

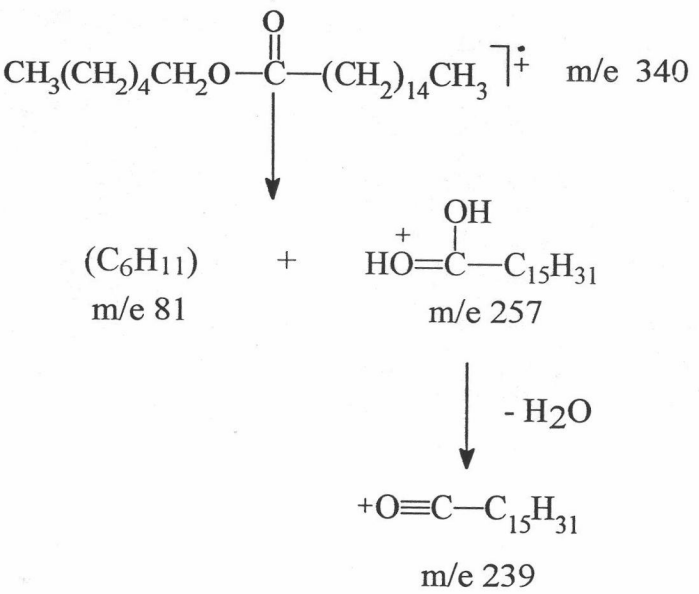
Figure A24, GC-chromatogram, indicated that hexyl ester product was composed of a mixture of hexyl ester of long chain fatty acid. The main

compositions were hexyl palmitate and hexyl oleate, at retention time 10.88 and 12.96, recpectively.

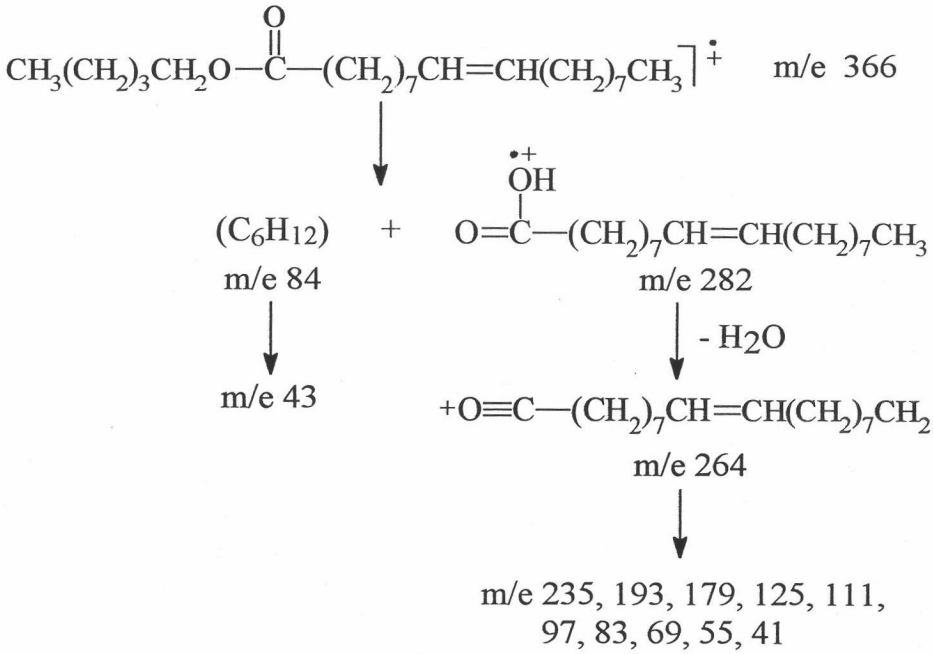
Mass-spectrum of hexyl palmitate (MW 340), at retention time 10.88, in Figure A25 showed base peak at 43 and m-256/e at 84 due to McLafferty rearrangement as shown in the following equation :



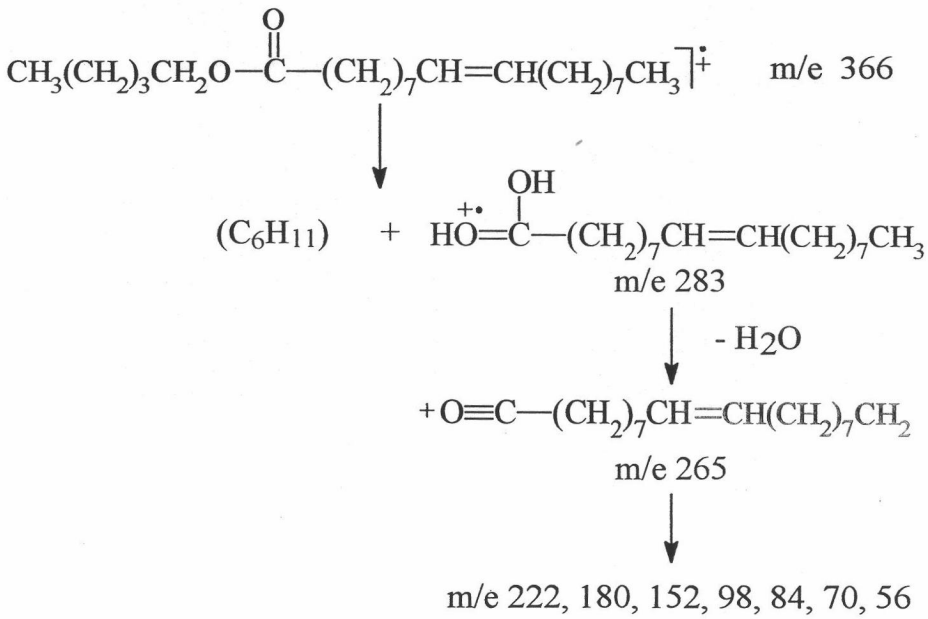
The reaction could occur as shown in the following equation :

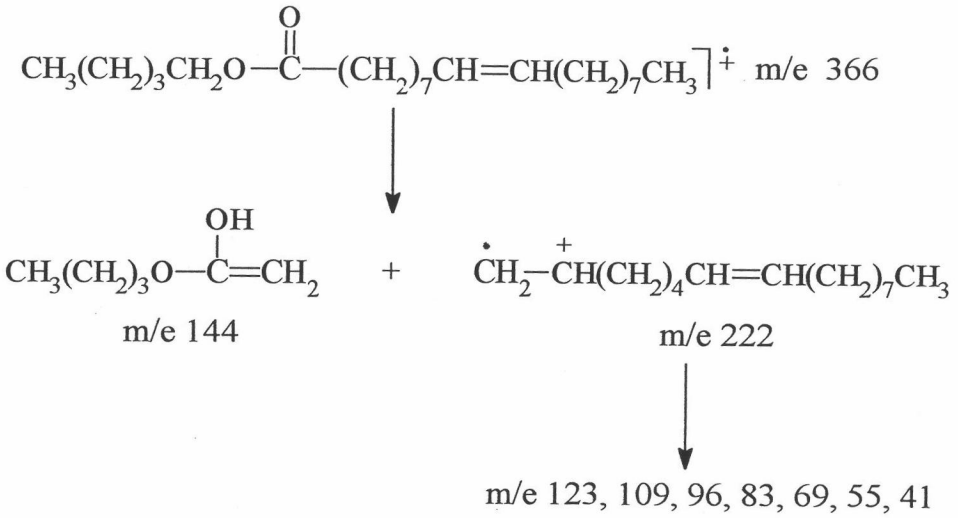


Mass-spectrum of hexyl oleate (MW 366), at retention time 12.96, in Figure A26 showed base peak at 43 due to McLafferty rearrangement as shown in the following equation :



The reaction could occur as shown in the following equation :





The physical and chemical properties of hexyl ester product, as showed in Table 4-2, were studied as follow : color, pour point, kinematic viscosity at 40 and 100 ^{O}C, viscosity index, flash point, and oxidation stability. The oxidation and thermal stability curve were analyzed by TGA analyzer and the results were shown in Figure A 43.

Table 4-2 : The physical and chemical properties of palm oil and hexyl ester product.

Properties	Palm Oil	Hexyl Ester Product
Color, ASTM	1.0	L2.5
Kinematic Viscosity		
@ 40 ^{O}C, cSt	40.26	7.12
@ 100 ^{O}C, cSt	8.37	2.40
Viscosity Index	190.48	183.86
Pour Point, ^{O}C	+12	-6
Flash Point, ^{O}C	314	230
Oxidation Point, ^{O}C	385	370
Oxidation Compounds, %wt	37.37	7.15

The result from Table 4-2 indicated that the pour point of product was -6 ^{O}C. Flash point was 230 ^{O}C. The viscosity at 40 and 100 ^{O}C were 7.12 and 2.40 cSt, respectively, and the viscosity index was 183.86. The color was lower than 2.5. The oxidative compounds were 7.15 %wt.

3. Transesterification of palm oil with cyclohexanol

The results of cyclohexyl ester from transesterification at optimum condition were shown by ^{13}C-NMR and GC-MS spectrum in Figure A10 and A30 , respectively.

From Figure A10, ^{13}C-NMR spectrum, when the reaction temperature was 90 ^{O}C and the reaction time was 3.5 hours, the result demonstrated that the peaks of triglyceride of palm oil at 62.02 and 68.86 ppm disappeared and the important peaks of —CH_2—O— and C=O of monoester product appeared at 72.1 and 173.1 ppm, recpectively. Inaddition, Figure A8 also showed peaks of unsaturated group at between 126 and 130 ppm.

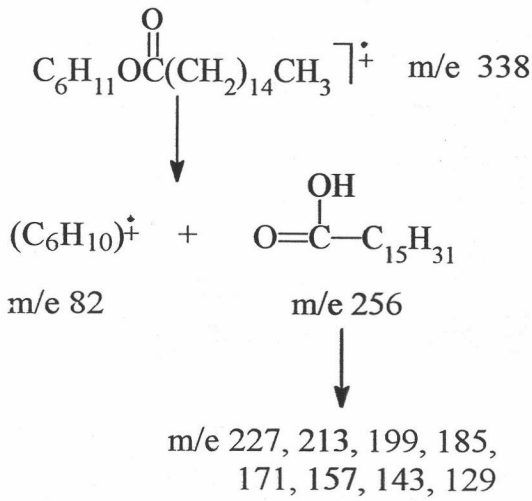
These experimental results indicated that the transesterification reaction of palm oil with cyclohexanol was complete at reaction temprature of 90 ^{O}C and reaction time of 3.5 hours. In this study, the yield of the resulting product was 91.17 %.

The composition of cyclohexyl ester product was determined by GC-MS. The GC-MS was performed in a DB-1 capillary column. The

GC-MS chromatogram was shown in Figure A30 and Mass spectrum of each peak was shown in Figure A31 to A32.

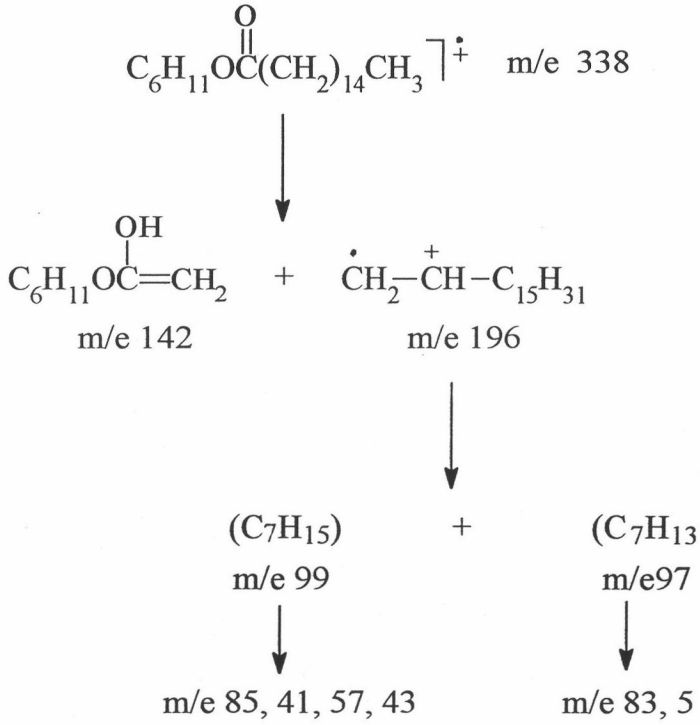
Figure A30, GC-chromatogram, indicated that cyclohexyl ester product was composed of a mixture of cyclohexyl ester of long chain fatty acid. The main compositions were cyclohexyl palmitate and cyclohexyl oleate, at retention time 11.66 and 13.83, recpectively.

Mass-spectrum of cyclohexyl palmitate (MW 338), at retention time 11.66, in Figure A31 showed base peak at 82 due to McLafferty rearrangement as shown in the following equation :

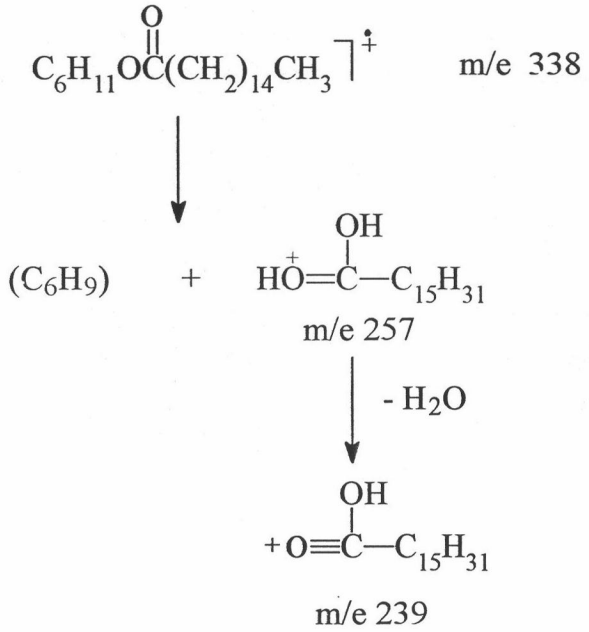


The reaction could occur as shown in the following equation (I and II) :

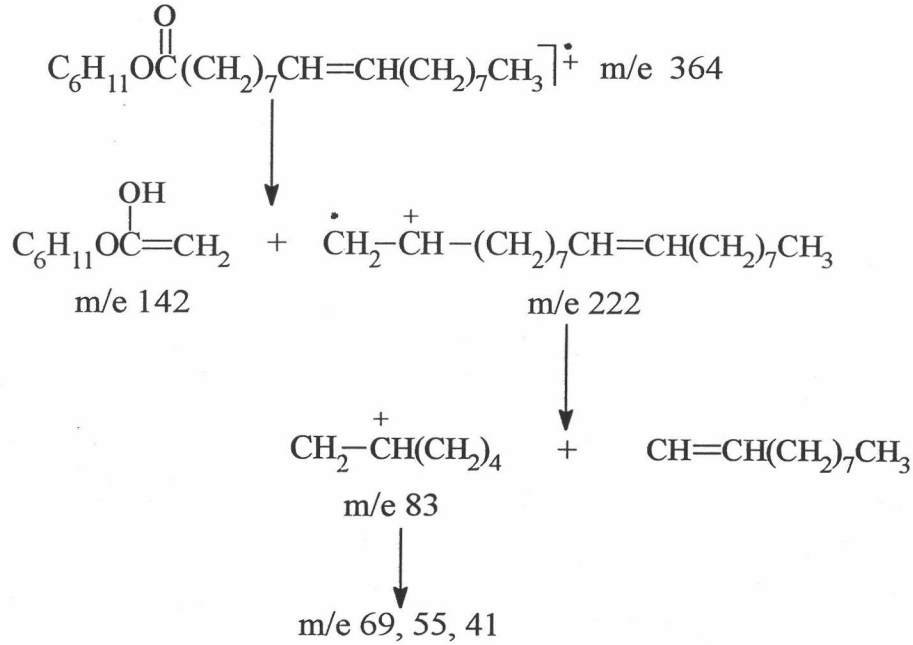
- Equation I : McLefferty rearrangement



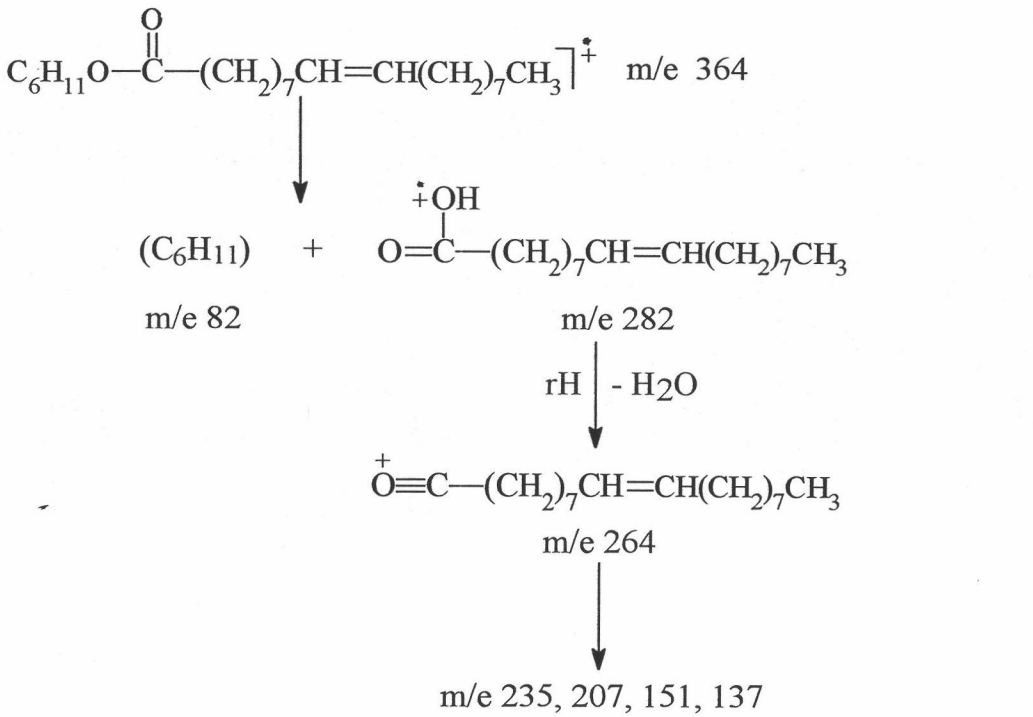
- Equation II : double H-shift



Mass-spectrum of cyclohexyl oleate (MW 364), at retention time 13.83, in Figure A 32 showed base peak at 55 and m-142/e at 222 due to McLafferty rearrangement as the following equation :



The reaction could occur as shown in the following equation :



The physical and chemical properties of cyclohexyl ester product, as showed in Table 4-3, were studied as follow : color, pour point, kinematic viscosity at 40 and 100 ^{O}C, viscosity index, flash point, and oxidation stability. The oxidation and thermal stability curve were analyzed by TGA analyzer and the results were shown in Figure A45.

Table 4-3 : The physical and chemical properties of palm oil and cyclohexyl ester product.

Properties	Palm Oil	Cyclohexyl Ester Product
Color, ASTM	1.0	2.5
Kinematic Viscosity		
@ 40 ^{O}C, cSt	40.26	11.99
@ 100 ^{O}C, cSt	8.37	3.29
Viscosity Index	190.48	154.44
Pour Point, ^{O}C	+12	-6
Flash Point, ^{O}C	314	232
Oxidation Point, ^{O}C	385	370
Oxidation Compounds, %wt	37.37	13.26

The result from Table 4-3 indicated that the pour point of product was -6 ^{O}C. Flash point was 232 ^{O}C. The viscosity at 40 and 100 ^{O}C were 11.99 and 3.29 cSt, respectively, and the viscosity index was 154.44. The color was 2.5. The oxidative compound was 13.26 %wt.

4. Transesterification of palm oil with 2-ethyl-1-hexanol

The results of 2-ethylhexyl ester from transesterification at optimum condition were shown by ^{13}C-NMR and GC-MS spectrum in Figure A12 and A36 , respectively.

From Figure A12, ^{13}C-NMR spectrum, when the reaction temperature was 80 ^{O}C and the reaction time was 1 hour, the result demonstrated that the peaks of triglyceride of palm oil at 62.02 and 68.86 ppm disappeared and the important peaks of —CH_2—O— and C=O of monoester product appeared at 66.4 and 173.7 ppm, recpectively. Inaddition, Figure A12 also showed peak of unsaturated group at between 127 and 130 ppm.

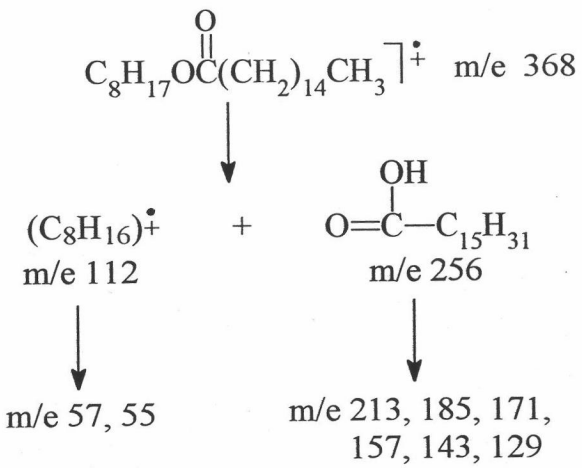
These experimental results indicated that the transesterification reaction of palm oil with 2-ethyl-1-hexanol was complete at the reaction temprature of 80 ^{O}C and the reaction time of 1 hour. In this study, the yield of the resulting product was 93.62 %.

The composition of 2-ethylhexyl ester product was determined by GC-MS. The GC-MS was performed in a DB-1 capillary column. The GC-MS chromatogram was shown in Figure A36 and Mass spectrum of each peak was shown in Figure A37 to A38.

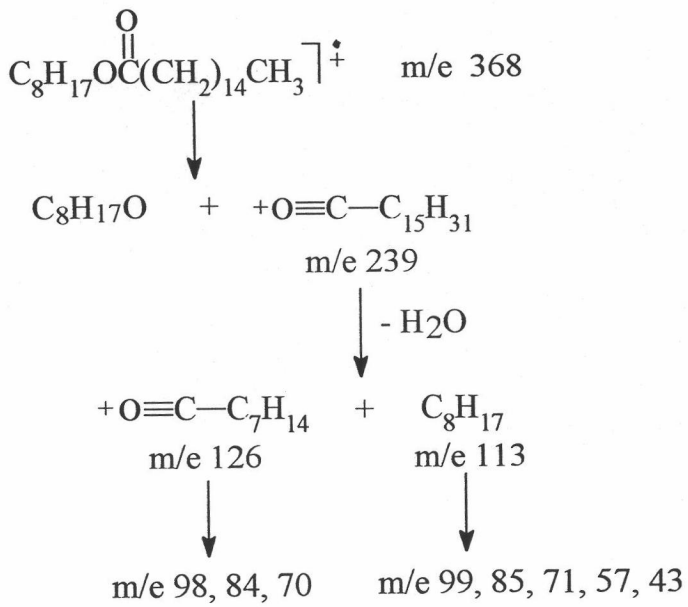
Figure A36, GC-chromatogram, indicated that 2-ethylhexyl ester product was composed of a mixture of 2-ethylhexyl ester of long chain fatty

acid. The main compositions were 2-ethylhexyl palmitate and 2-ethylhexyl oleate, at retention time 12.30 and 14.44, recpectively.

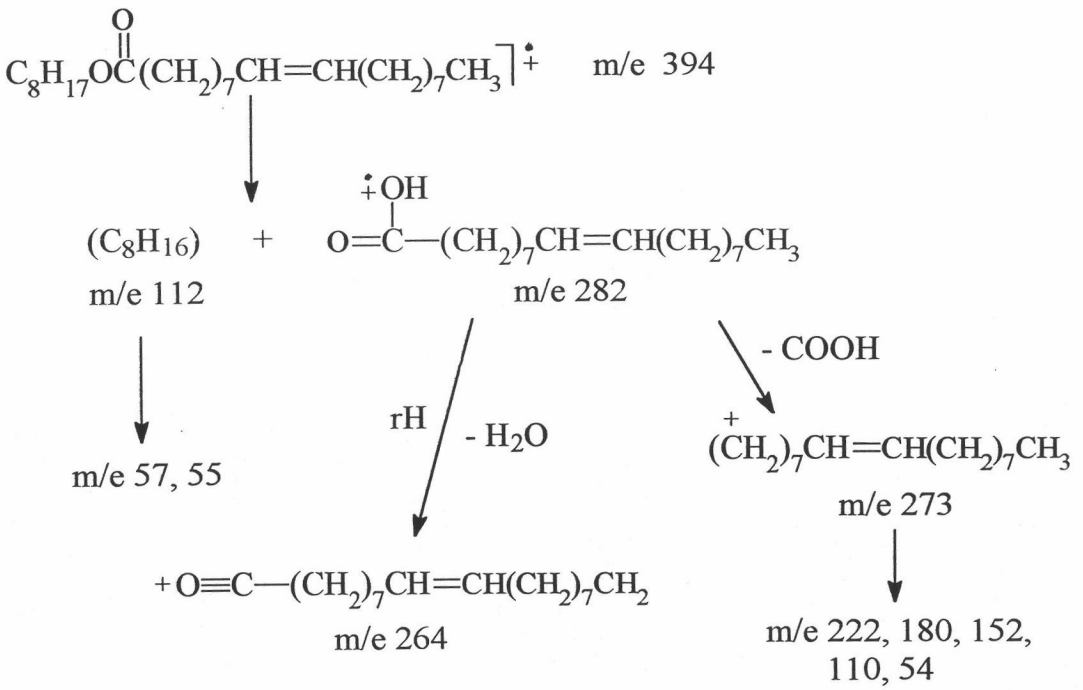
Mass-spectrum of 2-ethylhexyl palmitate (MW312), at retention time 12.30, in Figure A37 showed base peak at 112 due to McLafferty rearrangement as the following equation :



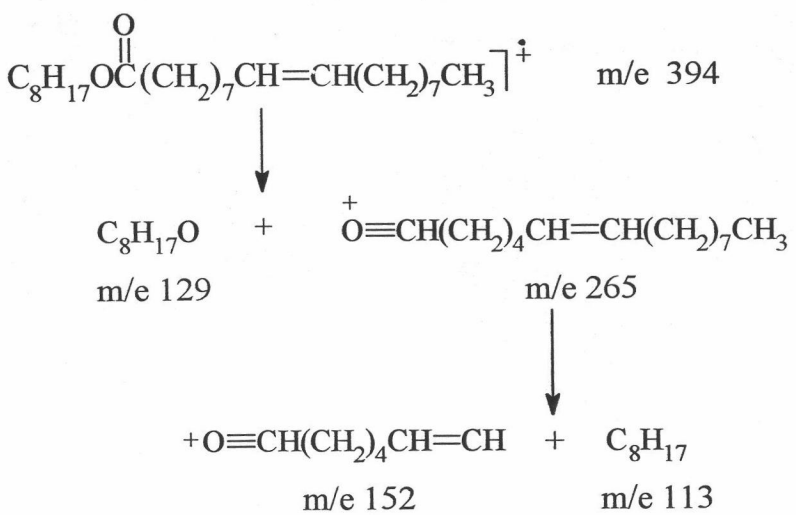
The reaction could occur as shown in the following equation :



Mass-spectrum of 2-ethylhexyl oleate (MW 394), at retention time 14.44, in Figure A37 shown base peak at 57 and m-282/e at 112 due to McLafferty rearrangement as the following equation :



The reaction could occur as shown in the following equation :



The physical and chemical properties of 2-ethylhexyl ester product, as showed in Table 4-4, were studied as follow : color, pour point, kinematic viscosity at 40 and 100 ^{O}C, viscosity index, flash point, and oxidation stability. The oxidation and thermal stability curve were analyzed by TGA analyzer and the result were showed in Figure A48.

Table 4-4 : The physical and chemical properties of palm oil and 2-ethylhexyl ester product.

Properties	Palm Oil	2-Ethylhexyl Ester Product
Color, ASTM	1.0	2
Kinematic Viscosity		
@ 40 ^{O}C, cSt	40.26	7.85
@ 100 ^{O}C, cSt	8.37	2.46
Viscosity Index	190.48	152.46
Pour Point, ^{O}C	+12	-12
Flash Point, ^{O}C	314	240
Oxidation Point, ^{O}C	385	375
Oxidation Compounds, %wt	37.37	9.36

The result from Table 4-4 indicated that the pour point of product was -12 ^{O}C. Flash point was 240 ^{O}C. The viscosity at 40 and 100 ^{O}C were 7.85 and 2.46 cSt, respectively and the viscosity index was 152.48. The color was 2. The oxidative compounds was 9.36%wt.

The color and oxidative compounds of monoester products, which resulted from unsaturated components, were still high comparing with petroleum base oil (Table A1). To improve the color and oxidation satability, the monoester had to be finally refined through hydrogenation or hydroxylation of unsaturated components.

Hydrogenation

The monoester products from transesterification reaction were treated with hydrogenated catalyst in a stirred autoclave batch reactor under hydrogen. The used catalyst contained 3% by weight of platinum supported on alumina which available commercially from United Catalyst Inc.,.

The hydrogenation process was optimized by varying the following parameters : hydrogen partial pressure, catalyst concentration , reaction time and reaction temperature while fixing other parameters, such as weight of oil and stirring speed. In this study, the hydrogen partial pressure was varied from 100 to 150 psi, the catalyst concentration was varied from 3, 4 and 5% by weight of oil, the reaction time was varied from 1 and 1.5 hours, and the reaction temperature was varied from 80 and 100 ^{O}C. The reaction was monitored by using ^{13}C-NMR.

Characteristics of hydrogenated ester products were determine by ^{13}C-NMR and GC-MS.

1. Hydrogenation of butyl ester

The results of hydrogenated butyl ester from hydrogenation at optimum condition was shown by ^{13}C-NMR and GC-MS spectrum in Figure A7 and A21 , respectively.

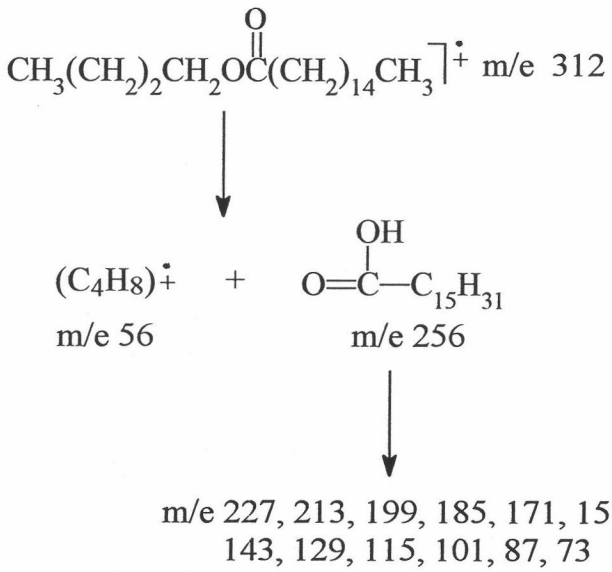
From Figure A7, ^{13}C-NMR spectrum, when hydrogen partial pressure was 120 psi, catalyst concentration was 4% by weight of oil, reaction time was 1.5 hours, and the reaction temperature was 100 ^{O}C, the result demonstrated that the peaks of unsaturated group at between 127 and 130 ppm disappeared but the important peaks of —CH_2—O— and C=O of butyl ester product still appeared at 63.8 and 173.5 ppm, recpectively.

These experimental results indicated that the hydrogenation reaction of butyl ester was completed at these reaction condition. In this study, the yield of the resulting product was 98.57 %.

The composition of hydrogenated butyl ester product was determined by GC-MS. The GC-MS was performed in a DB-1capillary column. The GC chromatogram was shown in Figure A21 and Mass spectrum of each peak were shown in Figure A22 to A23.

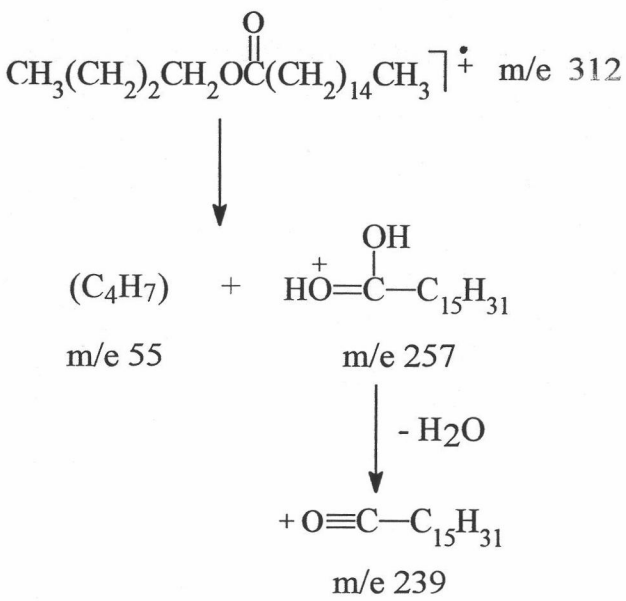
Figure A21, GC-chromatogram, indicated that hydrogenated butyl ester product was composed of a mixture of butyl ester of long chain fatty acid. The main compositions were butyl palmitate and butyl stearate, at retention time 8.82 and 10.93, recpectively.

Mass-spectrum of butyl palmitate (MW 312), at retention time 8.82, in Figure A22 showed base peak at 56 due to McLafferty rearrangement as shown in the following equation :

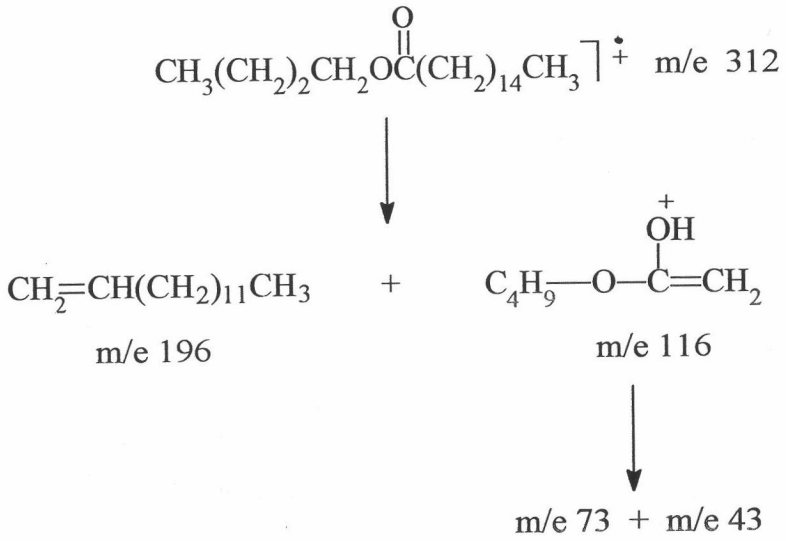


The reaction could occur by the following equation (I and II) :

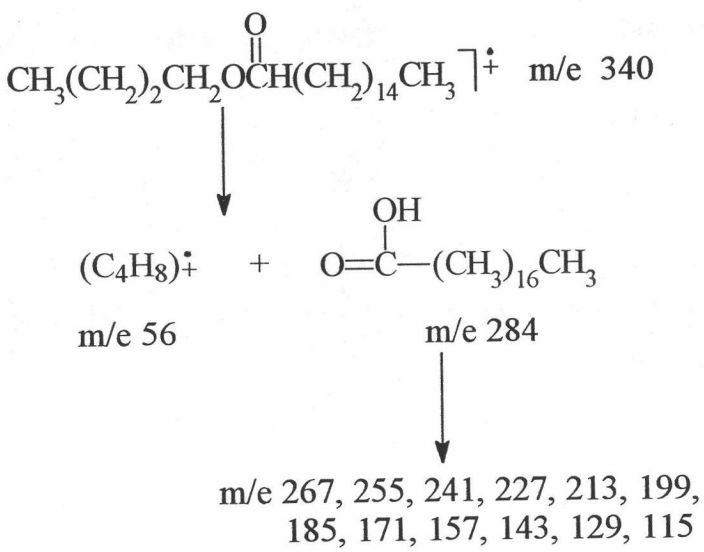
- Equation I : double H-shift



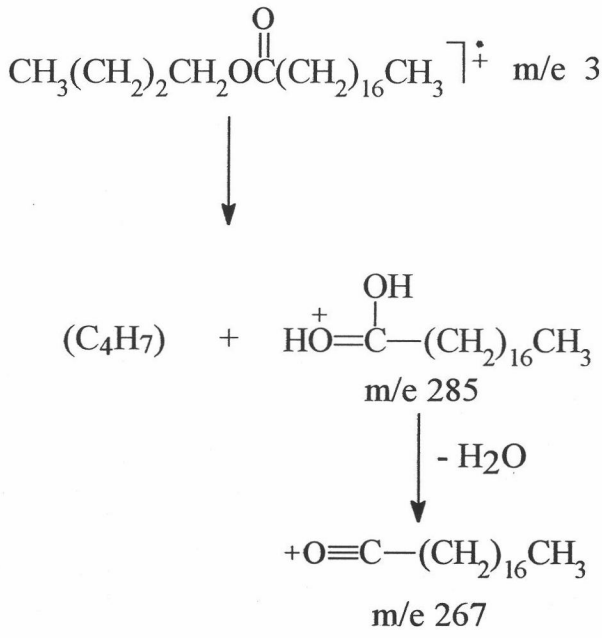
- Equation II : McLafferty rearrangement



Mass-spectrum of butyl stearate (MW340), at retention time 10.93, in Figure A23 showed base peak at 56 due to McLafferty rearrangement as shown in the following equation :



The reaction could occur by the following equation :



The physical and chemical properties of hydrogenated butyl ester product, as showed in Table 4-5, were studied as follow : color, pour point, kinematic viscosity at 40 and 100 ^{O}C, viscosity index, flash point, and oxidation stability. The oxidation and thermal stability curve were analyzed by TGA analyzer and the results were shown in Figure A42.

Table 4-5 : The physical and chemical properties of butyl ester product and hydrogenated butyl ester product

Properties	Butyl Ester Product	Hydrogenated Butyl Ester Product
Color, ASTM	L2	L1.5
Kinematic Viscosity		
@ 40 ^{O}C, cSt	6.04	6.77
@ 100 ^{O}C, cSt	2.26	2.31
Viscosity Index	197.04	181.84
Pour Point, ^{O}C	-6	+6
Flash Point, ^{O}C	215	213
Oxidation Point, ^{O}C	365	360
Oxidation Compounds, %wt	10.96	7.63

The result from Table 4-5 indicated that the pour point of product was +6 ^{O}C. Flash point was 213 ^{O}C. The viscosity at 40 and 100 ^{O}C were 6.77 and 2.31 cSt, respectively and the viscosity index was 181.84. The color was lower than 1.5. The oxidative compounds was 7.63 %wt.

2. Hydrogenation of hexyl ester

The results of hydrogenated hexyl ester from hydrogenation at optimum condition were shown by ^{13}C-NMR and GC-MS spectrum in Figure A9 and A27 , respectively.

From Figure A9, ^{13}C-NMR spectrum, when hydrogen partial pressure was 120 psi, catalyst concentration was 4% by weight of oil, reaction time was

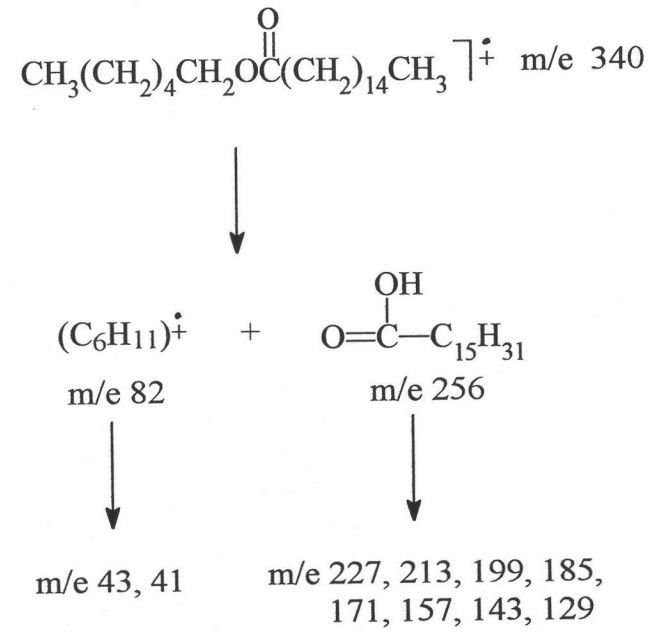
1.5 hours, and the reaction temperature was 100 ^{O}C, the result demonstrated that the peaks of unsaturated group at between 126 and 130 ppm disappeared but the important peaks of —CH_2—O— and C=O of butyl ester product still appeared at 63.8 and 173.5 ppm, recpectively.

These experimental results indicated that the hydrogenation reaction of hexyl ester was completed at these conditions. In this study, the yield of resulting product was 97.35 %.

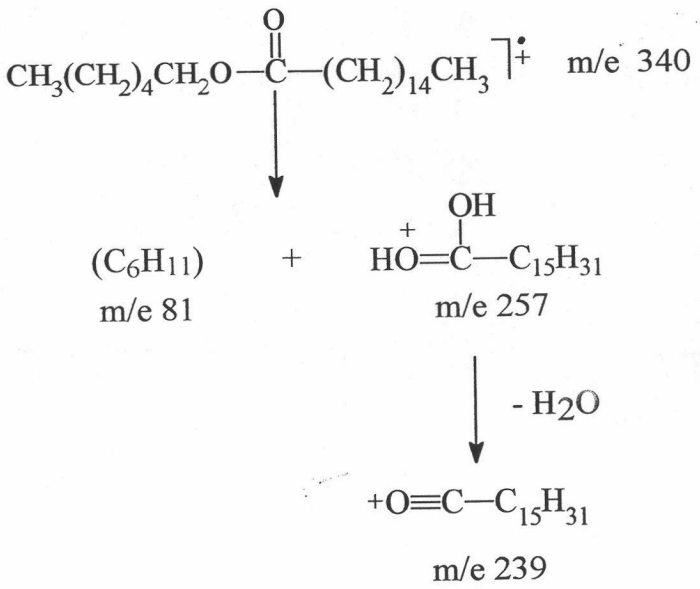
The composition of hydrogenated hexyl ester product was determined by GC-MS. The GC-MS was performed in a DB-1 capillary column. The GC-MS chromatogram was shown in Figure A27 and Mass spectrum of each peak were shown in Figure A28 to A29.

Figure A27, GC-chromatogram, indicated that hydrogenated hexyl ester product was composed of a mixture of hexyl ester of long chain fatty acid. The main compositions were hexyl palmitate and hexyl stearate, at retention time 10.73 and 13.15, recpectively.

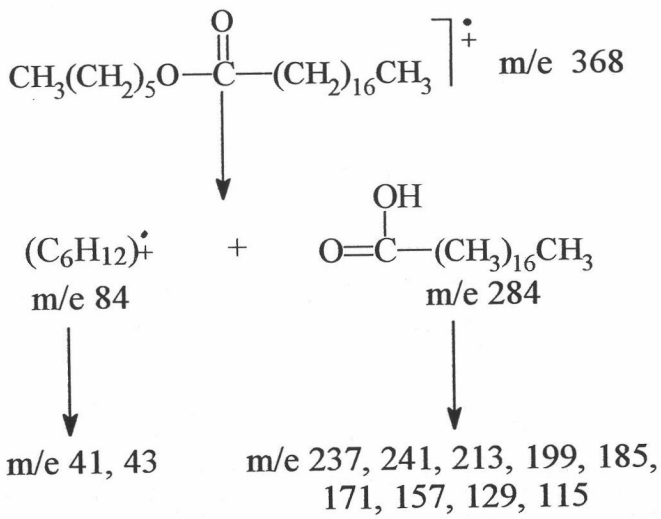
Mass-spectrum of hexyl palmitate (MW340), at retention time 10.73, in Figure A28 showed base peak at 43 due to McLafferty as show in the following equation :



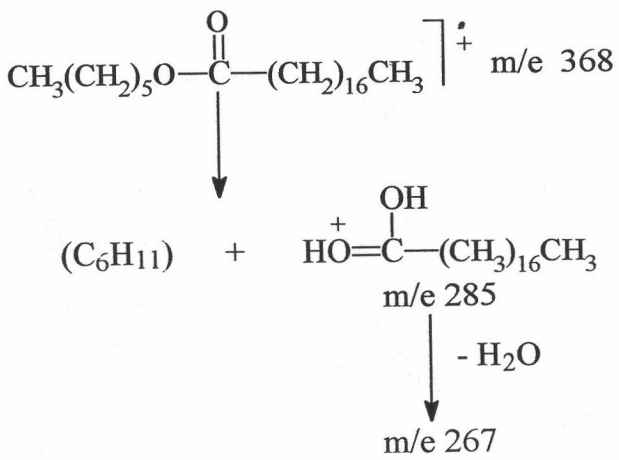
The reaction could occur by the following equation :



Mass-spectrum of hexyl stearate (MW 368), at retention time 13.15, in Figure A29 showed base peak at 43 and m-282/e at 84 due to McLafferty rearrangement the following equation :



The reaction could occur by the following equation :



The physical and chemical properties of hydrogenated hexyl ester product, as showed in Table 4-6, were studied as follow : color, pour point, kinematic viscosity at 40 and 100 ^{O}C, viscosity index, flash point, and

oxidation stability. The oxidation and thermal stability curve were analyzed by TGA analyzer and the results were shown in Figure A44.

Table 4-6 : The physical and chemical properties of hexyl ester product and hydrogenated hexyl ester product

Properties	Hexyl Ester Product	Hydrogenated Hexyl Ester Product
Color, ASTM	L2.5	L2
Kinematic Viscosity		
@ 40 ^{O}C, cSt	7.12	8.04
@ 100 ^{O}C, cSt	2.40	2.58
Viscosity Index	183.88	170.30
Pour Point, ^{O}C	-6	+6
Flash Point, ^{O}C	230	226
Oxidation Point, ^{O}C	370	370
Oxidation Compounds, %wt	7.15	7.83

The results from Table 4-6 indicated that the pour point of product was +6 ^{O}C. Flash point was 226 ^{O}C. The viscosity at 40 and 100 ^{O}C were 8.04 and 2.58 cSt, respectively and the viscosity index was 170.30. The color was 2. The oxidative compounds was 7.83 %wt.

3. Hydrogenation of cyclohexyl ester

The results of hydrogenated cyclohexyl ester from hydrogenation at optimum condition was shown by ^{13}C-NMR and GC-MS spectrum in Figure A11 and A33 , respectively.

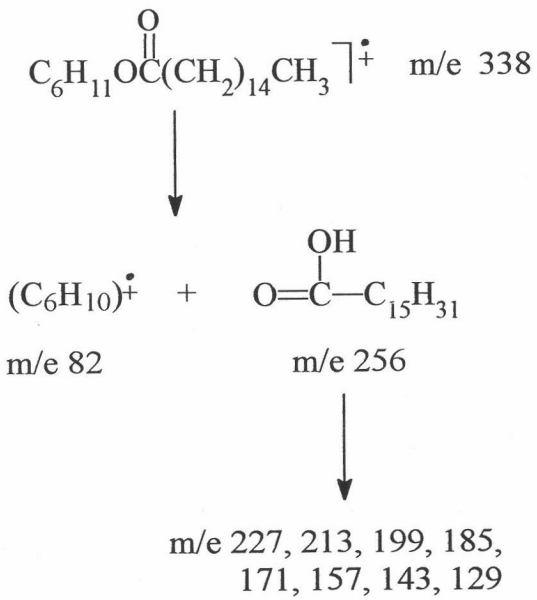
From Figure A11, ^{13}C-NMR spectrum, when hydrogen partial pressure was 120 psi, catalyst concentration was 4% by weight of oil, reaction time was 2 hours, and the reaction temperature was 100 ^{O}C, the result demonstrated that the peaks of unsaturated group at between 126 and 130 ppm disappeared but the important peaks of —CH_2—O— and C=O of butyl ester product still appeared at 63.8 and 173.5 ppm, recpectively.

These experimental results indicated that the hydrogenation reaction of cyclohexyl ester was completed at these condition. In this study, the yield of the resulting product was 97.65 %.

The composition of hydrogenated cyclohexyl ester product was determined by GC-MS. The GC-MS was performed in a column packed with DB-1. The GC-MS chromatogram was shown in Figure A33 and Mass spectrum of each peak were shown in Figure A34 to A35

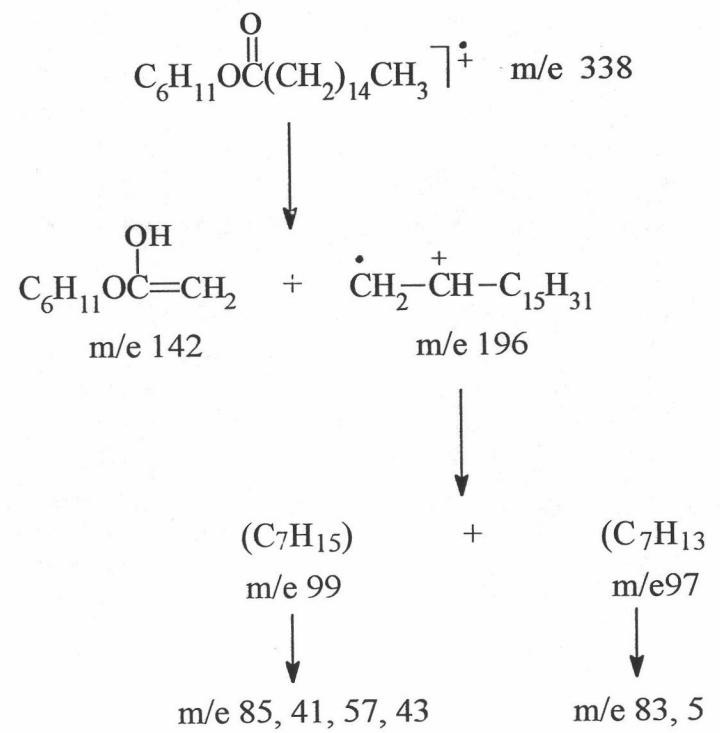
Figure A33, GC-chromatogram, indicated that hydrogenated cyclohexyl ester product was composed of a mixture of cyclohexyl ester of long chain fatty acid. The main compositions were cyclohexyl palmitate and cyclohexyl stearate, at retention time 11.77 and 14.29, recpectively.

Mass-spectrum of cyclohexyl palmitate (MW 338), at retention time 11.77, in Figure A34 showed base peak at 43 and m-142/e at 196 due to McLafferty rearrangement as the following equation :

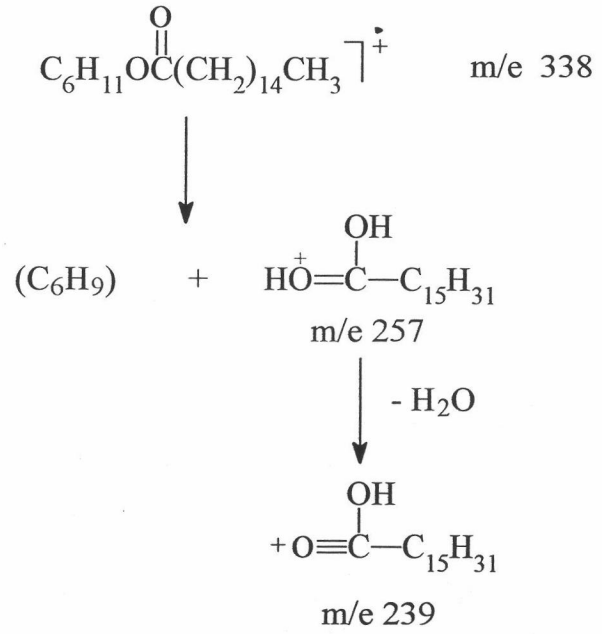


The reaction could occur by the following equation (I and II) :

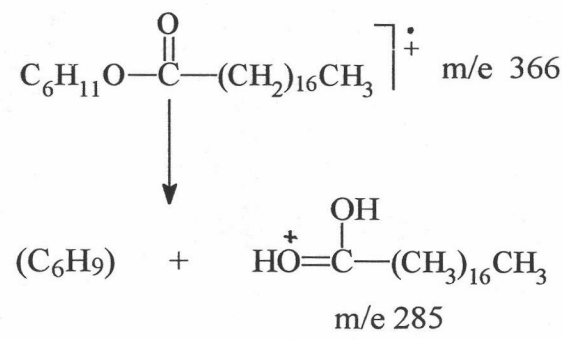
- Equation I : McLefferty rearrangement



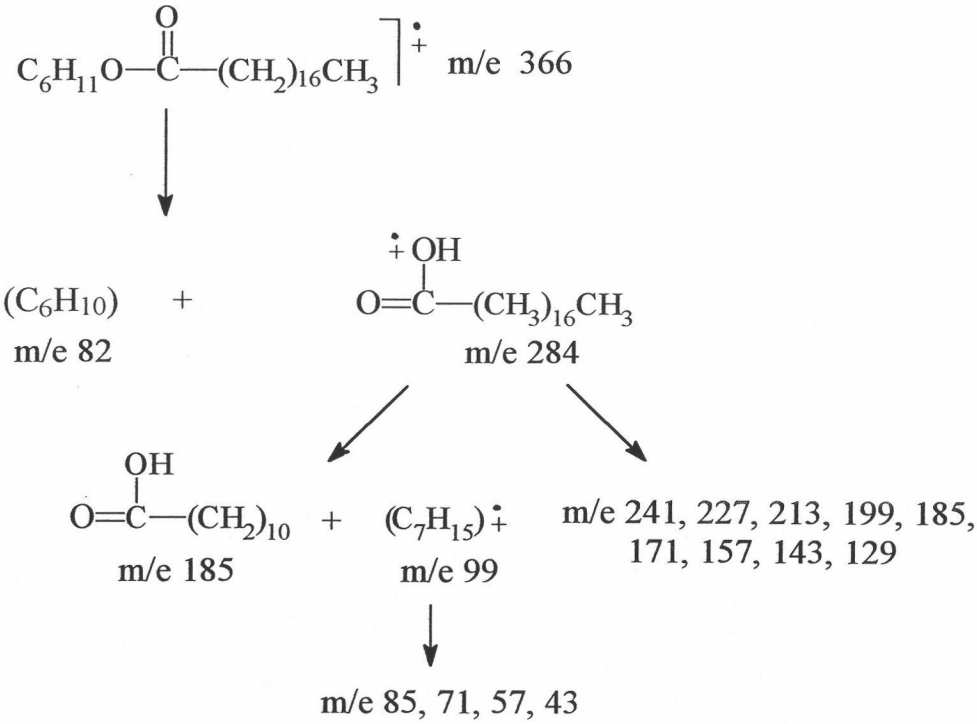
- Equation II : double H-shift



Mass-spectrum of cyclohexyl stearate (MW 366), at retention time 14.29, in Figure A35 showed base peak at 285 due to double H-shift as shown by the following equation :



The reaction could occur by the following equation :



The physical and chemical properties of hydrogenated cyclohexyl ester product, as showed in Table 4-7, were studied as follow : color, pour point, kinematic viscosity at 40 and 100 ^{O}C, viscosity index, flash point, and oxidation stability. The oxidation and thermal stability curve were analyzed by TGA analyzer and the sesult were shown in Figure A47.

Table 4-7 : The physical and chemical properties of cyclohexyl ester product and hydrogenated cyclohexyl ester product

Properties	Cyclohexyl Ester Product	Hydrogenated Cyclohexyl Ester Product
Color, ASTM	2.5	L2.5
Kinematic Viscosity		
@ 40 ^{o}C, cSt	11.99	13.56
@ 100 ^{o}C, cSt	3.29	3.48
Viscosity Index	154.44	149.03
Pour Point, ^{o}C	-6	+9
Flash Point, ^{o}C	232	227
Oxidation Point, ^{o}C	370	360
Oxidation Compounds, %wt	13.26	9.86

The result from Table 4-7 indicated that the pour point of product was +9 ^{o}C. Flash point was 227 ^{o}C. The viscosity at 40 and 100 ^{o}C were 13.56 and 3.48 cSt, respectively and the viscosity index was 149.03. The color was lower than 2.5. The oxidative compounds was 9.86 %wt.

4. Hydrogenation of 2-ethylhexyl ester

The results of hydrogenated 2-ethylhexyl ester from hydrogenation at optimum condition was shown by ^{13}C-NMR and GC-MS spectrum in Figure A13 and A39, respectively.

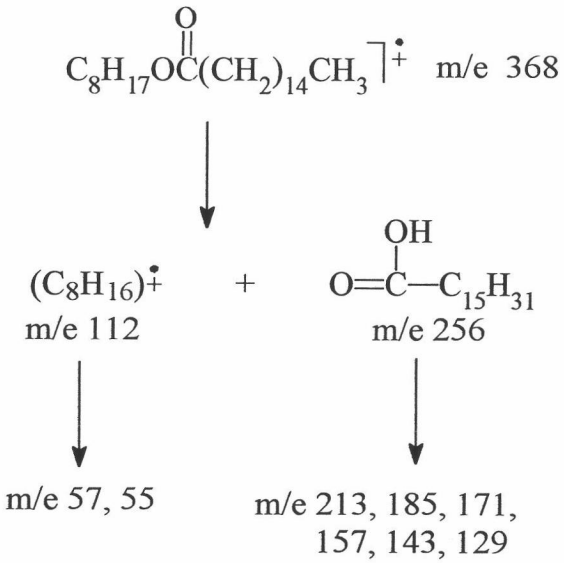
From Figure A13, ^{13}C-NMR spectrum, when hydrogen partial pressure was 120 psi, catalyst concentration was 4% by weight of oil, reaction time was 2 hours, and the reaction temperature was 100 ^{O}C, the result demonstrated that the peaks of unsaturated group at between 126 and 130 ppm disappeared but the important peaks of —CH_2—O— and C=O of 2-ethylhexyl ester product still appeared at 63.8 and 173.5 ppm, recpectively.

These experimental results indicated that the hydrogenation reaction of 2-ethylhexyl ester was completed at these condition. In this study, the yield of the resulting product was 98.73 %.

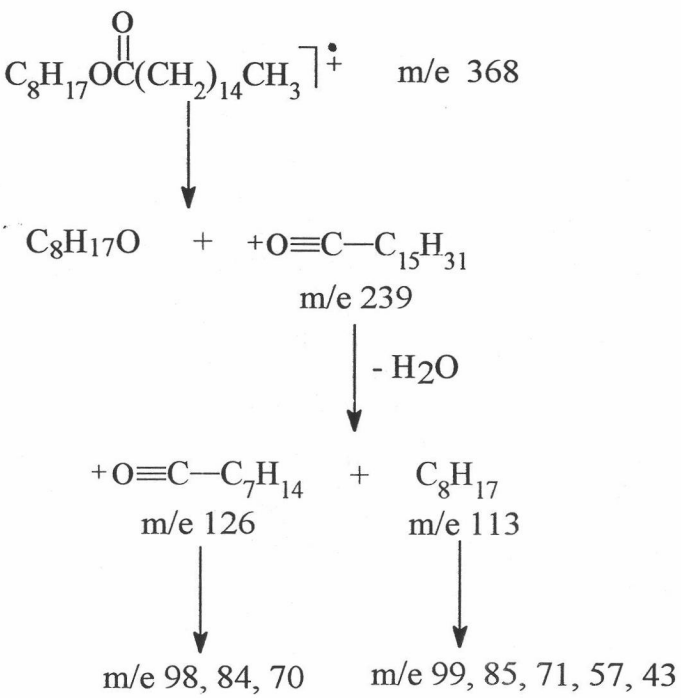
The composition of hydrogenated 2-ethyhexyl ester product was determined by GC-MS. The GC-MS was performed in a DB-1 capillary column. The GC chromatogram was shown in Figure A39 and Mass spectrum of each peak were shown in Figure A40 to A41

Figure A39, GC-chromatogram, indicated that hydrogenated 2-ethylhexyl ester product was composed of a mixture of 2-ethylhexyl ester of long chain fatty acid. The main composition were 2-ethylhexyl palmitate and 2-ethylhexyl stearate, at retention time 12.30 and 14.82, recpectively.

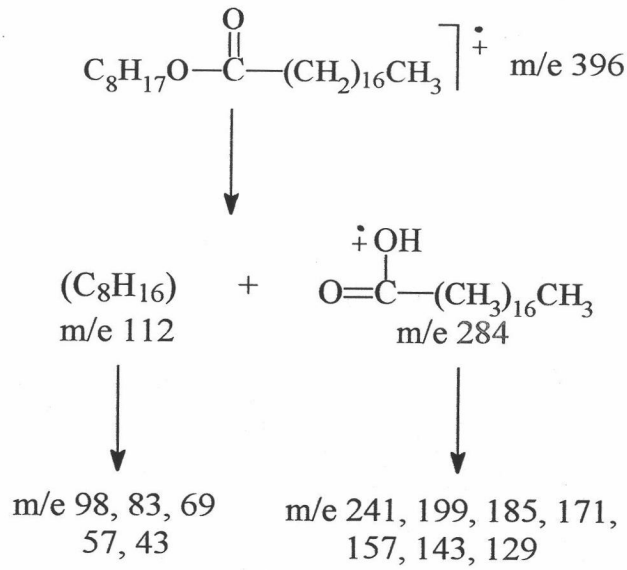
Mass-spectrum of 2-ethylhexyl palmitate (MW 368), at retention time 12.30, in Figure A40 showed base peak at 112 due to McLafferty rearrangement as show by the following equation :



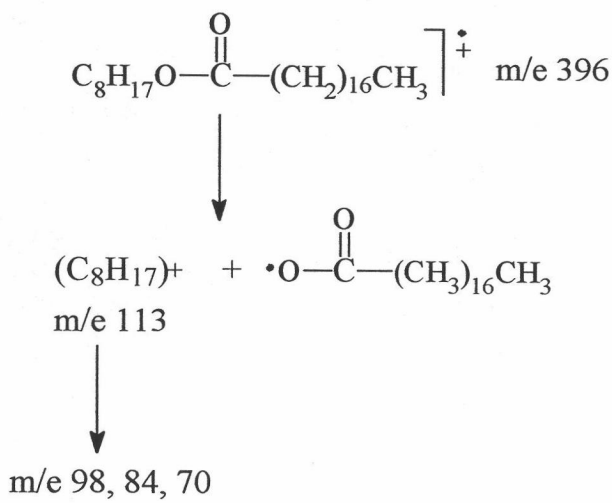
The reaction could occur by the following equation :



Mass-spectrum of 2-ethylhexyl stearate (MW 396), at retention time 14.82, in Figure A41 showed base peak at 112 due to McLafferty rearrangement as shown by the following equation :



The reaction could occur by the following equation :



The physical and chemical properties of hydrogenated 2-ethylhexyl ester product, as showed in Table 4-8, were studied as follow : color, pour point, kinematic viscosity at 40 and 100 ^{O}C, viscosity index, flash point, and oxidation stability. The oxidation and thermal stability curve were analyzed by TGA analyzer and the result were shown in Figure A49.

Table 4-8 : The physical and chemical properties of 2-ethylhexyl ester product and hydrogenated 2-ethylhexyl ester product

Properties	2-Ethylhexyl Ester Product	Hydrogenated 2-Ethylhexyl Ester Product
Color, ASTM	2	1.5
Kinematic Viscosity		
@ 40 ^{O}C, cSt	7.58	9.24
@ 100 ^{O}C, cSt	2.46	2.75
Viscosity Index	152.46	156.39
Pour Point, ^{O}C	-12	+3
Flash Point, ^{O}C	240	238
Oxidation Point, ^{O}C	375	360
Oxidation Compounds, %wt	9.36	5.92

The result from Table 4-8 indicated that the pour point of product was +3 ^{O}C. Flash point was 238 ^{O}C. The viscosity at 40 and 100 ^{O}C were 9.24 and 2.75 cSt, respectively and the viscosity index was 156.39. The color was 1.5. The oxidative compounds was 5.92 %wt.

Hydroxylation of 2-ethylhexyl ester

The 2-ethylhexyl ester from transesterification reaction were treated by hydroxylation. These process consists of two distinct processes : peroxidation of carbon-carbon double bonds and acid-catalyzed cleavage of epoxides.

The results of epoxydation products from peroxidation of carbon-carbon double bonds at optimum condition was shown by ^{13}C-NMR in Figure A14.

From Figure A14, ^{13}C-NMR spectrum, when reaction temperature was 20 ^{O}C and reaction time was 4 hours, the result demonstrated that the peaks of carbon-carbon double bonds of 2-ethylhexyl ester between 127 and 130 ppm disappeared and the important peaks of epoxide appeared at 57.49 and 57.45 ppm, recpectively.

These experimental results indicated that the peroxidation of carbon-carbon double bonds of 2-ethylhexyl ester was completed at reaction temprature of 20 ^{O}C and reaction time of 4 hours.

The results of 2-ethylhexyl-9,10-hydroxy ester products from acid catalyzed cleavage of epoxides at optimum condition was shown by ^{13}C-NMR and GC-MS spectrum in Figure A15 and A42, respectively.

From Figure A15, ^{13}C-NMR spectrum, when reaction temperature was 60 ^{O}C and reaction time was 2 hours, the result demonstrated that the peaks of epoxide of 2-ethylhexyl ester between 57.49 and 57.45 ppm disappeared and

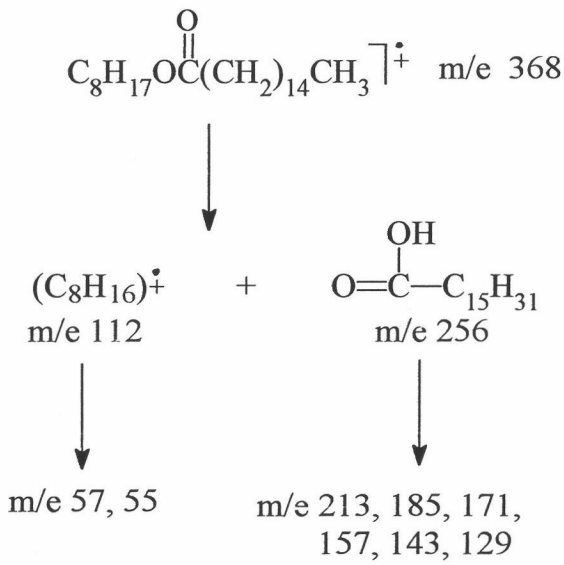
the important peaks of -OH group appeared at 72.38 and 74.51 ppm, recpectively.

These experimental results indicated that the acid-catalyzed cleavage of epoxides was completed at reaction temperature of 60 ^{O}C and reaction time of 2 hours. In this study, the yield of the resulting product was 72.52%.

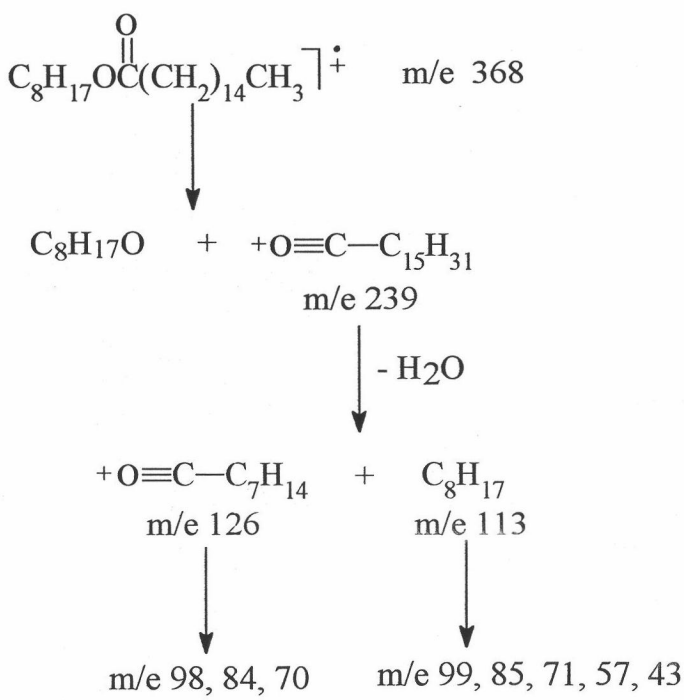
The composition of 2-ethylhexyl-9,10-hydroxy ester product was determined by GC-MS. The GC-MS was performed in a DB-1 capillary column. The GC chromatogram was shown in Figure A42 and Mass spectrum of each peak were shown in Figure A43 to A44

Figure A42, GC-chromatogram, indicated that hydrogenated 2-ethylhexyl ester product was composed of a mixture of 2-ethylhexyl ester of long chain fatty acid. The main compositions were 2-ethylhexyl palmitate and 2-ethylhexyl-9,10-hydroxyl stearate, at retention time 12.30 and 14.82, recpectively.

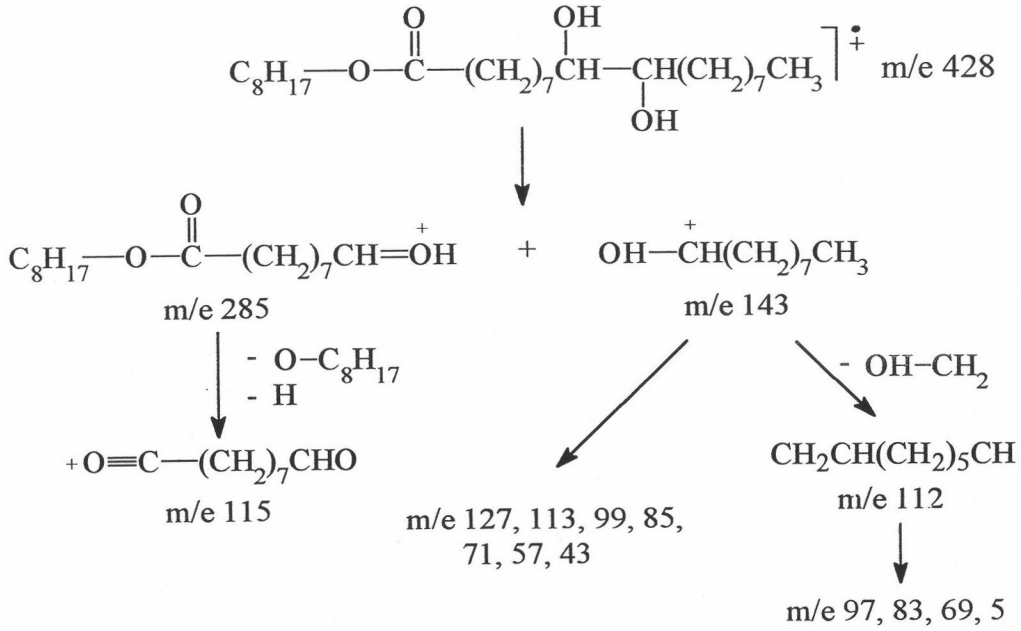
Mass-spectrum of 2-ethylhexyl palmitate (MW 368), at retention time 12.30, in Figure A43 showed base peak at 112 due to McLafferty rearrangement as shown by the following equation :



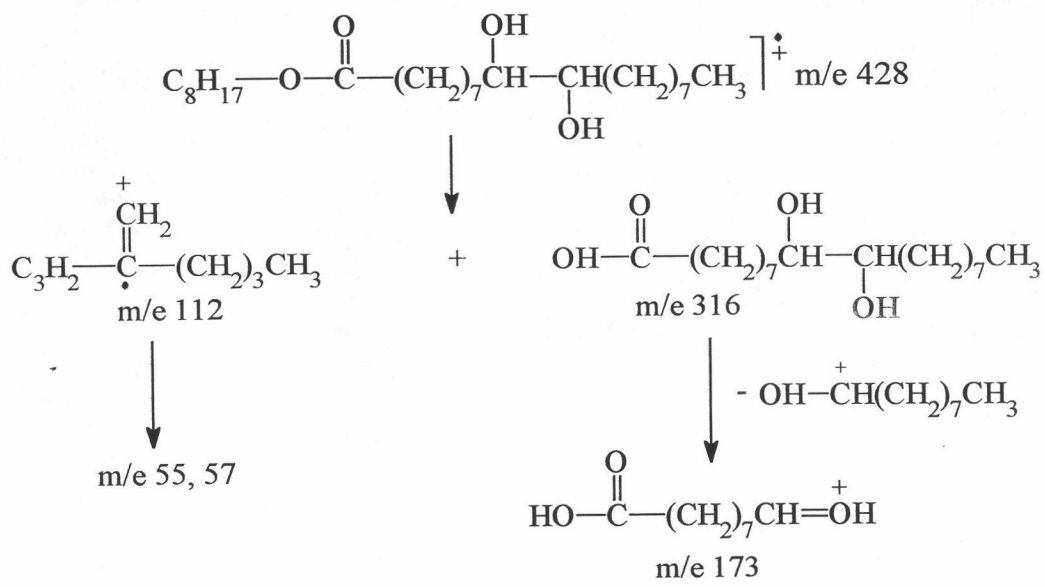
The reaction could occur by the following equation :



Mass-spectrum of 2-ethylhexyl-1,9-hydroxyl stearate (MW 428), at retention time 19.57, in Figure A44 showed base peak at 155 due to McLafferty rearrangement as shown by the following equation :



The reaction could occur by the following equation :



The physical and chemical properties of ester product, as showed in Table 4-8, were studied as follow : color, pour point, kinematic viscosity at 40 and 100 ^{O}C, viscosity index, flash point, and oxidation stability. The oxidation and thermal stability curve were analyzed by TGA analyzer and the result were shown in Figure A50.

Table 4-8 : The physical and chemical properties of 2-ethylhexyl ester product and hydrogenated 2-ethylhexyl ester product

Properties	2-Ethylhexyl Ester Product	2-Ethylhexyl Ester Product from Hydroxylation
Color, ASTM	2	1.5
Kinematic Viscosity		
@ 40 ^{O}C, cSt	7.58	9.24
@ 100 ^{O}C, cSt	2.46	2.75
Viscosity Index	152.46	156.39
Pour Point, ^{O}C	-12	+3
Flash Point, ^{O}C	240	238
Oxidation Point, ^{O}C	375	375
Oxidation Compounds, %wt	9.36	5.92

The results from Table 4-8 indicated that the pour point of product was +3 ^{O}C. Flash point was 238 ^{O}C. The viscosity at 40 and 100 ^{O}C were 9.24 and 2.75 cSt, respectively and the viscosity index was 156.39. The color was 1.5. The oxidative compounds was 5.92 %wt.

Lubricating base oil (150SN) blended with butyl ester

This process studied ability of hydrogenated ester as VI improver for lubricating base oil(150SN). This studied used hydrogenated butyl ester to blend with 150SN since hydrogenated butyl ester has the highest VI. The physical properties of 150SN blended with hydrogenated butyl ester, were shown in Table 4-9.

The results from Table 4-9 indicated that the viscosity index of blended oils increase from 103 to 135. The pour point was increased from -9 to -3 ^{O}C. The viscosity at 40 ^{O}C was decreased from 30 to 17 cSt and the viscosity at 100 ^{O}C decreased from 5 to 4 cSt. The color was 1.5. Flash point of was 238 ^{O}C. The oxidative compounds was 5.92 %wt.

Table 4-9 : The properties of lube base oil (150SN) and lube base oil blended with butyl ester.

Properties	Lube base oil (150SN)	Butylester in composition (%)									
		3	6	9	12	15	18	21	24	27	30
Color, ASTM	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	L1.0	L1.0	L1.0
Pour Point, °C	-9	-9	-9	-6	-6	-3	-3	0	0	+3	+3
Kinematic Viscosity											
@ 40 °C	30.54	27.83	26.43	24.67	22.73	21.74	20.56	19.37	18.45	17.22	16.29
@ 100 °C	5.25	5.05	4.96	4.79	4.59	4.49	4.37	4.25	4.15	3.99	3.88
Viscosity Index	103.85	108.45	112.56	115.94	118.59	120.11	123.14	126.72	130.12	132.57	134.94
Oxidation Point, °C	313.2	-	-	-	-	-	-	-	313.20	-	-
Oxidation	17.61	-	-	-	-	-	-	-	-17.61	-	-
Compounds, %wt											