CHAPTER 3
EXPERIMENTAL

Materials

1. Coconut oil
   commercial grade;
2. 1-Hexanol
   analytical grade; Fluka
3. 1-Octanol
   analytical grade; Fluka
4. 2-Ethyl-1-hexanol
   analytical grade; Fluka
5. Cyclohexanol
   analytical grade; Fluka
6. Sulphuric acid (98% W/W)
   analytical grade ; Rhone-Poulenc
7. Diethyl ether
   reagent grade : J.T.Baker Inc.
8. Sodium sulfate anhydrous
   reagent grade ; Fluka
9. Sodium hydrogen carbonate
   reagent grade ; Fluka
10. 3%Pt on alumina
    United Catalyst Inc.
11. Hydrogen gas (purity 99.5% minimum)
    industrial gas ; T.I.G. Trading Ltd.

Apparatus and Instrument

1. Fourier-Transform NMR spectrometer :
   Model AC-F200 (200 MHz), Bruker Spectrospin
2. Fourier-Transform IR spectrometer :
   Model 1760X, Perkin Elmer
3. Gas Chromatography-Mass Spectrometer :
   Model MD-800 equipped with a 800 series GC, Fison Instruments
4. High pressure reactor : Consisting of high pressure batch stirred autoclave :
   model 4561 with temperature controller model 4841, Parr Instrument Company
5. Colourimeter :
   The Fisher ASTM (D 1500)
6. Viscometer :
   Model K-234 A, Hocher Instrument Co., Inc.
7. Pour point Tester :
   Model A82, HAKKE
8. Flash Point Tester :
   Model Cleveland semi-automatic
9. Thermogravimetric Analyzer :
   Model STA 490 C, Netzsch

Procedures

1. Transesterification of coconut oil

Coconut oil 30g. and 1-hexanol 27 ml.(excess)(1) were mixed together in 250 ml round-bottomed flask equipped with magnetic stirrer, and concentrated sulphuric acid 5 ml (5%by volume of alcohol) was poured in the mixture. The reaction flask was fitted with condensor and thermometer. The mixture was heated at approximately 70°C(2) with continuous stirring for 3-6 hours. The reaction was stopped by dropping 2-3 drops of water into the reaction mixture and then allowed cool to room temperature. The excess acid was neutralized with saturated sodium bicarbonate solution. The mixture neutralized was dissolved in diethyl ether and separated with distilled water to remove glycerol by using 500 ml separatory funnel. After removal of the aqueous layer, the organic layer was dried with anhydrous sodium sulfate. Next diethyl ether was evaporated and excess alcohol was removed by distillation under reduced pressure. The yield of monoester product was determined by weighting.

NOTE: 1) The alcohol was varied from 1-hexanol, 1-octanol, 2-ethyl-1-hexanol, cyclohexanol, respectively.

2) The temperature was varied from 70°C, 80°C, 90°C, respectively for selecting the optimum condition.

2. Determination

2.1 Color,ASTM	by ASTM D 1500
2.2 Kinematic viscosity	by ASTM D 445
2.3 Viscosity index	by ASTM D 2270
2.4 Pour point	by ASTM D 97
2.5 Flash point	by ASTM D 92

2.6 The oxidation point and the percentages of oxidative compounds were determined by TGA method under oxidation atmosphere.

TGA condition

Heating rate	:	5° C
Temperature range	:	20-700° C
Atmosphere	:	O_2
Flow rate	:	50

3. Hydrogenation process

The monoester 300g obtained by transesterification procedure, was poured into the reactor and catalyst was added. The reactor was closed and spilt ring closures were moved into its position and cap screws were tighted with the bomb in the reactor.

Oxygen gas was removed by fludging with hydrogen gas. After fludging for 2-3 minutes, a gas release valve was closed. The regulator was adjusted to the desired pressure and then the valve of hydrogen tank and gas inlet valve were closed, respectively.

Next, a thermocouple was inserted into a sturdy thermowell attached to the underside of the reactor head and extened to a point near the bottom of the reactor cavity followed by connecting the stirring motor and pass water into the cooling channel.

The desired temperature was setted by at the temperature controller. The heater was switched on. The stirring speed was adjusted about 175 rpm.

The reaction was allowed to occur for the specific time before the motor and heater were switched off to stop the reaction. After a gas release valve was opened, the pressure was reduced to atmospheric pressure. Stirring motor was disconnected when the reactor temperature reactor reach 50°c. A thermowell was pulled out of the reactor head and the reactor was opened. The hydrogenated oil was poured in a beaker. Finally, the catalyst was seperated from the mixture by filtering with a Whatman paperfilter No.1. The characteristics of hydrogenated oil were determined by ^{13}C-NMR and GC-MS.

Note: 1) The reaction temperature was varied at 100, 150°c

2) The H_2 pressure was varied at 70, 100, 150 psi

3) The reaction time was varied at 3, 6 hours

4) The concentration of catalyst (%by wt. of oil) was varied at 1, 3, 5

4. Determination

4.1 Color,ASTM	by ASTM D 1500
4.2 Kinematic viscosity	by ASTM D 445

4.3 Viscosity index by ASTM D 2270

4.4 Pour point by ASTM D 97

4.5 Flash point by ASTM D 92

4.6 The oxidation point and the percentages of oxidative compounds were determined by TGA method under oxidation atmosphere.

TGA condition

Heating rate : 5° C

Temperature range : 20-700° C

Atmosphere : O_2

Flow rate : 50

GC-MS condition

Column : DB-5 Capillary column
30 x 0.25 ID x 0.25 μm

Carrier gas : Helium , 40 mm/sec

Oven : 80 to 200 ° C(2 min) rate 25 ° C/min
200 to 275 ° C (2 min) rate 5° C/min

Injection temperature : 250° C

Detector : MS(EI^+ 70) ev