CHAPTER 3

EXPERIMENTAL

3.1 Apparatus and Instruments

1. Hydrogenator

   Parr Instrument Company, model 4561 Pressure reactor with 4841 Temperature controller

2. Fourier-Transform NMR Spectrometer

   Bruker, model AC-F 200

3. Colorimeter

   Fisher, ASTM D1500

4. Viscosity Apparatus

   Hochler Instrument Co.,Inc., model K-234A

5. Pour Point Apparatus

   HAAKE, model A82

6. Flash Point Apparatus

   PERZOO, model Cleveland semi-automatic

7. Thermogravimetric Analyzer

   Shimadzu, model TGA-30

8. Gas Chromatograph-Mass Spectrometer

   Fisons Instruments, model MD-800 equipped with a 8000 series GC

3.2 Materials

1. Castor oil

   medicinal grade ; Vidhyasom Co.,Ltd.

2. 2-Ethyl-1-hexanol

   analytical grade ; Fluka

3. Sulphuric acid (98% w/w)

   analytical grade ; Rhone-Poulenc

4. Diethyl ether

   reagent grade ; J.T.Baker Inc.

5. Sodium sulfate anhydrous

   reagent grade ; Fluka

6. 3% Pt on alumina

   United Catalyst Inc.

7. Hydrogen gas (purity 99.5% minimum)

   industrial grade ; T.I.G. Trading Ltd.

3.3 Procedures

3.3.1 Transesterification of castor oil with 2-ethyl-1-hexanol

Reagent was prepared by dissolving concentrated sulphuric acid (5ml, 5% by volume of alcohol) in 2-ethyl-1-hexanol (100 ml, 83.3 g. 0.64 mole) contained in a 250-ml erlenmeyer flask. This reagent was added into castor oil (100 g, 0.107 mole) contained in a 500-ml round-bottomed flask fitted with a condenser and magnetic stirrer. The mixture was heated at about 60^{o}C(1) with

continuous stirring for 3-6 hours. The reaction mixture was allowed to cool at room temperature and then was extracted with ether and distilled water in a 500-ml separatory funnel. The etheral layer was washed with water until neutral to litmus paper. After removal of the aqueous layer, the organic layer was dried with anhydrous sodium sulfate. Then the ether was evaporated and the excess alcohol was distilled under reduced pressure. The yield of monoester product was determined by weighing. In this case, the yield was 112.73 g (85.66%yield). The characteristics of monoester product were determined by ^{13}C-NMR and GC-MS.

Note : (1) The temperature was varied from 60°C to 80, 100, 130, and 160°C respectively to select a suitable reaction condition.

3.3.2 Determination of physical and chemical properties of monoester product as follow :

Physical properties

1.	Color	by ASTM D1500
2.	Kinematic viscosity	by ASTM D445
3.	Viscosity index	by ASTM D2270
4.	Pour point	by ASTM D97
5.	Flash point	by ASTM D92/93

Chemical properties

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

2. The middle point of thermal stability curve that show thermal stability function were determined by TGA method under N_2 atmosphere.

3.3.3 Hydrogenation Process

The prepared ester (130 g) was charged into the reactor and the required quantity of catalyst was added. The reactor was then closed and split ring closures were moved into the position from the sides and cap screws were tighted with the bomb in the heater.

In order to remove all oxygen gas in the reactor, a gas inlet valve and a gas release valve had to be open as well as a valve of hydrogen tank. The regulator was also adjusted until pressure gauge indicated 10 psi. After charging for 2 minutes, a gas release valve was closed. The regulator was adjusted to the desired pressure and then the valve of hydrogen tank and a gas inlet valve were closed respectively.

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

The desired temperature was setted at the temperature controller. The heater was switched on. The stirring speed was adjusted to 300 rpm.

The reaction was allowed to occur for the specific time before the motor and the 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 reached 50^{o}C. A thermowell was pulled out of the bomb head and the reactor was opened. The hydrogenated oil was poured in to a beaker. Finally, the catalyst was separated from the mixture by vacuum filtering with a Whatman filter No. 1. The yield was

120.55 g(92.73% yield). The characteristics of hydrogenated oil were determined by ^{13}C-NMR and GC-MS.

3.3.4 Selecting a suitable operating condition

The effect of hydrogen partial pressure, catalyst concentration, reaction time and reaction temperature were studied under conditions which were shown in table 3.1.

3.3.5 Determination of physical and chemical properties of hydrotreated oil as follow :

Physical properties

1. Color by ASTM D1500
2. Kinematic viscosity by ASTM D445
3. Viscosity index by ASTM D2270
4. Pour point by ASTM D97
5. Flash point by ASTM D92/93

Chemical properties

1. The oxidation point and percentages of oxidative compounds were determined by TGA method under air atmosphere.
2. The middle point of thermal stability curve that show thermal stability function were determined by TGA method under N_2 atmosphere.

Table 3.1 The various operating conditions for the experiment at a constant agitation 300 rpm

parameters Studied	Temperature (^{o}C)	Concentration of catalyst (%by wt. of oil)	Times (hrs.)	H_2 pressure (psi)
Pressure	100	3	3	100
	100	3	3	300
Catalyst concentration	100	1	3	100
	100	2	3	100
	100	3	3	100
	100	4	3	100
Reaction time	100	4	2	100
	100	4	3	100
	100	4	4	100
	100	4	5	100
Temperature	100	4	3	100
	80	4	3	100