

CHAPTER 3

EXPERIMENTAL

Chemical

1. Soybean oil (commercial grade) was obtained from a market.
2. Lubricating base oil (150 SN) was obtained from Nippon Kaiji Kentei Kyokai
3. 2-Ethyl-1-hexanol (analytical grade) , 1-hexanol (analytical grade) , 4-methyl-2-pentanol (analytical grade) and 1- butanol (analytical grade) were obtained from Fluka
4. Isopropyl alcohol (analytical grade) was obtained from Carlo Erba reagent (RPE)
5. Sulfuric acid (98%; analytical grade) and diethyl ether (reagent grade) were obtained from J.T. Baker
6. Acetic acid (99.8%; glacial) was obtained from Riedel-de Haen AG.
7. Hydrogen peroxide (30 Gew.%), sodium sulfate anhydrous (reagent grade) , hexachloroplatinic acid (Pt 40%) and sodium hydrogen carbonate (reagent grade) were obtained from Fluka
8. Hydrogen gas (purity 99.5% minimum; industrial grade) was obtained from T.I.G. Trading Ltd.
9. Alumina support catalyst was obtained from United Catalysts Inc.

Instruments

1. Fourier-Transform NMR Spectrometer : Model AC-F200 (200 MHz), Bruker Spectrospin
2. Fourier-Transform IR Spectrometer : Model 1760X, Perkin Elmer
3. Gas Chromatograph - 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 and a temperature controller model 4841, Parr Instrument Company
5. Colorimeter : The Fisher ASTM (D 1500)
6. Viscometer : Model K-234 A, Hoher 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

Experimental Procedure

1. Transesterification and Hydrogenation of Soybean Oil [19,20]

1.1 Transesterification

The concentrated sulfuric acid (5% by volume of alcohol) and quantities of alcohol , the alcohol was varied by using 148 ml (5 mol) of 1-Butanol, 182 ml (4.5 mol) of 1-Hexanol, 228 ml (4.5 mol) of 2-ethyl-1-hexanol, 255 ml (5.5 mol) of 4-methyl-2-pentanol and 149 ml (6.5 mol) of isopropyl alcohol , were mixed in 500 ml two-necked glass round-bottomed flask. Then, equipped with a magnetic stirrer, and stirred until mixture was dissolved. Soybean oil 300 g was added into the reaction flask which was fitted with a condenser and a thermometer. The flask was placed in an oil-bath for heating to 80°C . The reaction temperature was varied at 70, 80 and 90 °C to select a suitable reaction with continuous stirring for 1-4 hours. After that, the reaction mixture was allowed to cool at room temperature and neutralize the excess acid with saturated sodium bicarbonate solution. After the sodium bicarbonate solution was separated by using a 500 ml separatory funnel, the reaction mixture was washed with distilled water, and diethyl ether may be added to help good extraction. After removal of the aqueous part, the organic layer was dried over anhydrous sodium sulfate. The excess alcohol was removed under reduced pressure. The yield of monoester product was determined by weighing.

The characteristics of monoester products were analyzed by ^{13}C -NMR

After transesterification, the product was determined the following properties :

1. Color by ASTM D 1500
2. Kinematics viscosity by ASTM D 445
3. Viscosity index by ASTM D 2270
4. Pour point by ASTM D 97
5. Flash point by ASTM D 92
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 ml / min.

1.2 Hydrogenation

The monoesters which were obtained from experimental procedure 1 at optimum operating were changed into the stainless steel reactor with the 1% platinum on alumina. Then, closed the reactor . The split ring closure were moved into the position from the sides and the cap screws were tighten. The gas in reactor was replaced by feeding hydrogen gas, and a gas

release valve was opened. After charged for 2 to 3 minutes, a gas release valve was closed, and then hydrogen gas was fed until the pressure of the reactor reached working pressure.

The reactor was placed in the heating unit, and a thermocouple was inserted into a thermowell. Water was passed into the cooling channel. A power bottom was switch on. The desired reaction temperature, and reaction time were setted at the controller. The motor stirring and heater were switched on, and then the stirring speed was adjusted to 370 rpm.

After the reaction took place, the heater was switched off to stop the reaction. When the reaction temperature reached 50°C, a thermocouple was pulled out, and the motor stirring was stopped. Then a gas release valve was opened, and the pressure was reduced to atmospheric pressure. Finally, the hydrogenated oil was transferred to a beaker, and the catalyst was separated from the mixture by filtering with a Whatman filter No.1 or centrifuging. The yield of hydrogenated oil was determined by weighting.

The characteristics of monoester products were analyzed by ^{13}C -NMR and GC-MS

GC-MS condition

Column : DB-1 Capillary column
30 x 0.25 ID x 0.25 μm

Carrier gas : Helium, 40 mm/sec

Oven : 90 to 180 ° C(4 min.) rate 12 ° C/min.
180 to 250 ° C (6 min.) rate 15° C/min.

Injection temperature : 250° C

Detector : MS(EI⁺ 70) eV

After hydrogenation, the product was determined the following properties:

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|---|----------------|
| 1. Color | by ASTM D 1500 |
| 2. Kinematics viscosity | by ASTM D 445 |
| 3. Viscosity index | by ASTM D 2270 |
| 4. Pour point | by ASTM D 97 |
| 5. Flash point | by ASTM D 92 |
| 6. Total Acid Number (TAN) | by ASTM D 974 |
| 7. Copper Strip Corrosion | by ASTM D 130 |
| 8. 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 ml / min.

2. Blending

The hydrogenated monoester which received from procedure 3.3.2 was weighed 9 g (3% by weight of blended oil) and mixed the monoester with 291 g of lube base oil number SN150 into a beaker. The mixture was stirred until homogeneously. The blended oil was transferred to the bottle. Prepared the varied qualities of the blended at 3, 5, 8, 13, 18 and 20 % in the procedure which described previously. Each sample was determined in the following properties .

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|-------------------------|----------------|
| 1. Color | by ASTM D 1500 |
| 2. Kinematics viscosity | by ASTM D 445 |
| 3. Viscosity index | by ASTM D 2270 |
| 4. Pour point | by ASTM D 97 |