

CHAPTER III

EXPERIMENTAL

3.1 Chemicals

1. Palm oil (commercial grade) was obtained from Morakot Industry Co.,Ltd..
2. Oleic acid (analytical grade) was obtained from Fluka.
3. Stearic acid (analytical grade) was obtained from Fluka.
4. Palmitic acid (analytical grade) was obtained from Fluka.
5. Lubricating base oil (150SN) was obtained from Inchcape Testing Service (S) Pte Ltd..
6. Methanol (analytical grade) was obtained from Fluka.
7. 1,3-propanediol (analytical grade) was obtained from Fluka.
8. 1,4-butanediol (analytical grade) was obtained from Fluka.
9. 1,5-pentanediol (analytical grade) was obtained from Fluka.
10. 2,2-dimethyl-1,3-propanediol (analytical grade) was obtained from Fluka.
11. 2-ethyl-1,3-hexanediol (analytical grade) was obtained from Fluka.
12. Sulfuric acid (98% ; analytical grade) was obtained from J.T.Baker.
13. Diethyl ether (reagent grade) was obtained from J.T.Baker.
14. Sodium sulfate anhydrous (reagent grade) was obtained from Fluka.
15. Toluene (analytical grade) was obtained from Fluka.

3.2 Apparatus and Instruments

1. Fourier-Transform NMR spectrometer : Model AC-F200 (200 MHz), Bruker spectrosprin
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. Colorimeter ; The Fisher ASTM (D1500)
5. Viscometer : Model K-234 A, Hochler Instrument Co.,Inc.
6. Pour point Tester : Model A82, HAKKE
7. Flash point Tester : Model Cleveland semi-automatic
8. Thermogravimetric Analyzer : Model STA 490C, Netzsch

3.3 Experimental Procedures

3.3.1 Transesterification of Palm Oil with Methanol

- Procedure In the transesterification process, concentrated sulfuric acid (3% by volume of alcohol) was dissolved in methanol (25 ml, 19.75 g, 0.62 mole) contained in a 500-ml two necked glass round- bottomed flask, equipped with magnetic stirrer. The mixture was stirred until it was homogeneous, then 100 g (0.118 mole) of palm oil was added and the flask was placed in an oil-

bath at various temperatures (70, 80 or 90°C) with continuous stirring for 3-6 hours. The reaction mixture was allowed to cool to room temperature and then neutralized with saturated sodium bicarbonate solution. The mixture was dissolved in diethyl ether and extracted with distilled water (to remove glycerol) in a 500-ml separatory funnel. After removal of the aqueous layer, the organic layer was dried with anhydrous sodium sulfate. Next the diethyl ether and the excess methanol were evaporated. The yield of methyl ester product was determined by weight.

- Determination of chemical and physical properties of methyl ester product as follows :

Chemical properties

1. The characteristics of the methyl ester was determined by FTIR, ^{13}C -NMR, and GC-MS.

GC-MS conditions

Column : DB-1 capillary column

30 m 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)

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

TGA conditions

Heating rate : 5 °C/min

Temperature range : Ambient to 650 °C

Atmosphere : Dynamic air 100 ml/min

Reference : Al₂O₃

Physical properties

1. Color, ASTM 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

3.3.2 Re-transesterification of Methyl Ester with Diols

- Procedure Concentrated sulfuric acid (1% by weight of diol) was mixed with 0.33 mole of diol (1,3-propanediol, 1,4-butanediol, 1,5-pentanediol, 2,2-dimethyl-1,3-propanediol, and 2-ethyl-1,3-hexanediol) This was added to the methyl ester 200 g (0.65 mole), obtained from experimental procedure 3.3.1, at the optimum operating conditions, contained in a 500-ml round-bottomed flask fitted with a distillation apparatus (If the methanol produced in

the reaction was removed by distillation) and a magnetic stirrer. The mixture was heated at various temperatures (70, 80, and 90 °C) to select a suitable reaction with continuous stirring for 3-6 hours. The reaction mixture was allowed to cool at room temperature and then was neutralized with saturated sodium bicarbonate solution. The mixture was dissolved in diethyl ether and extracted with distilled water in a 500-ml separatory funnel. After removal of the aqueous layer, the organic layer was dried with anhydrous sodium sulfate. Next the diethyl ether was evaporated. The yield of diester products were determined by weight.

- Determination of chemical and physical properties of diester products
as follow :

Chemical properties

1. The characteristics of diester products were determined by FTIR, ¹³C-NMR, and GC-MS.

GC-MS condition

Column : DB-1 capillary column

30 m 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)

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

TGA conditions

Heating rate : 5 °C/min

Temperature range : Ambient to 650 °C

Atmosphere : Dynamic air 100 ml/min

Reference : Al₂O₃

Physical properties

1. Color, ASTM 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

3.3.3 Esterification of Fatty Acids with Diols

- Procedure Concentrated sulfuric acid (1 % by weight of diol) was dissolved in 0.33 mole of diol (1,3-propanediol, 1,4-butanediol, 1,5-pentanediol, 2,2-dimethyl-1,3-propanediol, or 2-ethyl-1,3-hexanediol) and an azeotroping agent such as toluene. This mixture was added to 0.65 mole of fatty acid (oleic acid, stearic acid, or palmitic acid) contained in a 500-ml round-bottomed flask fitted with a Dean-Stark Apparatus, and magnetic stirrer.

The mixture was heated at various temperatures (110, 130, and 150 °C), to select a suitable conditions, with continuous stirring for 3-6 hours. After stopped the reaction, the reaction mixture was allowed to cool to room temperature and then was neutralized with saturated sodium bicarbonate solution. The mixture was dissolved in diethyl ether and extracted with distilled water in a 500-ml separatory funnel. After removal of the aqueous layer, the organic layer was dried with anhydrous sodium sulfate. Next the diethyl ether was evaporated and the toluene was removed by distillation under reduced pressure. The yield of diester products were determined by weight.

- Determination of chemical and physical properties of diester products
as follow :

Chemical properties

1. The characteristics of diester products were determined by FTIR, ¹³C-NMR, and GC-MS.

GC-MS condition

Column : DB-1 capillary column

30 m 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)

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

TGA conditions

Heating rate : 5 °C/min

Temperature range : Ambient to 650 °C

Atmosphere : Dynamic air 100 ml/min

Reference : Al₂O₃

Physical properties

1. Color, ASTM 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

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