CHAPTER III

EXPERIMENTAL

3.1 Chemicals

1. Lauric acid (analytical grade) was obtained from Vicchi International (Thailand) Co., Ltd.
2. Myristic acid (analytical grade) was obtained from Vicchi International (Thailand) Co., Ltd.
3. Palmitic acid (analytical grade) was obtained from Merck Ltd.
4. Stearic acid (analytical grade) was obtained from Fluka.
5. 1,2-ethanediol (analytical grade) was obtained from Carlo ERBA.
6. 1,2-propanediol (analytical grade) was obtained from Carlo ERBA.
7. Sulfuric acid (98%, analytical grade) was obtained from J.T.Baker.
8. Sodium Sulfate anhydrous (reagent grade) was obtained from MERCK Ltd.
9. Sodium Hydrogen carbonate (reagent grade) was obtained from Fluka.
10. Toluene (analytical grade) was obtained from J.T.Baker.
11. Base oil (500SN and 150SN) was obtained from the Petroleum Authority of Thailand

3.2 Apparatus and Instruments

1. Fourier - Transform NMR spectrometer: Model AC - F 200 (200 MHz) Bruker spectrospin
2. Fourier - Transform IR spectrometer: Model 1760X, Perkin Elmer
3. Thermogravimetric Analyzer: Model TGA 7, Perkin Elmer
4. Electrothermal: Model 9100
5. Automatic flash point tester: Model CFP 92 (COC), ISL
6. Total acid number tester: Model Metrohm 665
7. Colorimeter: The Fisher ASTM (D 1500)
8. Pour point tester: Model A82, HAKKE
9. Auto viscometer: Model V066230 (VH1), ISL
10. Foaming tester: Model Petrotest

3.3 Experimental Procedure

3.3.1 Preparation of diester products

The diester products were prepared from the reaction of glycol with fatty acid, i.e., 1,2-ethanediol and 1,2-propanediol, and fatty acid, i.e., lauric acid, myristic acid, palmitic acid and stearic acid. For example, the procedure for diesters synthesis was described below.

The reagent was prepared by dissolving concentrated sulfuric acid (1% by weight of glycol) in 0.3 mole of 1,2-ethanediol (excess 30%), and added 200 ml of toluene as azeotropic agent. This reagent was added into 0.6 mole of lauric acid which was contained in a 500 ml round - bottomed flask fitted with a Dean and Stark water separation unit. Then the mixture was heated by electromentle for 3-6 hours and the reaction temperature was about 110°C. The water was separated by forming the azeotrope with toluene. After the reaction was stopped, the reaction mixture was allowed to cool at room temperature. Then the mixture was added into a 500 ml separatory funnel and washed with distilled water. The organic layer was neutralized with saturated sodium hydrogen carbonate solution and washed again with distilled water. The organic layer was dried with anhydrous sodium sulfate. Finally toluene was distilled to obtain diester products.

3.3.2 Preparation of lubricants

Lubricants were prepared by blending base oil with a diester product, which the weight ratio of base oil to diester products were 120:3, 120:2 and 120:1. The base oil used were 150 SN and 500 SN. The process to prepare lubricants comprises the steps of heating the diester product and then added the diester to the base oil or directly dissolved the solid diester in heated base oil at a temperature higher than the melting point of the solid diester.

3.3.3 Determination of chemical properties of diester products.

3.3.3.1 Characteristics of diester products were determined by FTIR and ^{13}C - NMR.

3.3.3.2 The oxidation point, thermal stability and the percentages of oxidative compounds were determined by TGA method under air atmosphere.

TGA condition

Heating rate: 20°C / min

Temperature range: 50 to 450°C

Atmosphere: Air

3.3.3.3 The acid number was determined by ASTM D 974.

3.3.4 Determination of physical properties of diester products.

3.3.4.1 Melting point was determined by electrothermal instrument.

3.3.4.2 Color was determined by ASTM D 1500.

3.3.4.3 Flash point was determined by ASTM D 92.

3.3.5 Determination of physical properties of lubricants.

3.3.5.1Color was determined by ASTM D 1500.

3.3.5.2 Flash point was determined by ASTM D 92.

3.3.5.3 Pour point was determined by ASTM D 97.

3.3.5.4 Kinematic viscosity was determined by ASTM D 445.

3.3.5.5 Viscosity index was determined by ASTM D 2270.

3.3.5.6 Foaming test was determined by ASTM D 892.