

CHAPTER III

EXPERIMENTAL

3.1 Chemicals

1. 1-Butanol for Synthesis was obtained from MERCK.
2. 1-Hexanol for Synthesis was obtained from MERCK.
3. 1-Octanol for Synthesis was obtained from MERCK.
4. 2-Ethyl-1-hexanol for Synthesis was obtained from MERCK.
5. 4-Methyl-2-pentanol for Synthesis was obtained from MERCK.
6. Sodium sulfate anhydrous (Reagent grade) was obtained from CARLO ERBA.
7. Toluene (Reagent grade) was obtained from CARLO ERBA.
8. Sulfuric acid 98 % (Reagent grade) was obtained from CARLO ERBA.
9. Sodium hydrogen carbonate (Reagent grade) was obtained from MERCK
10. Diethyl ether (Reagent grade) was obtained from J.T. Baker

3.2 Apparatus and Instruments

1. Seta-Lovibond Color Comparator : Model 1525, STANHOPE SETA
2. Automatic PMCC Flash Point Tester : Model MP-329/330, WALTER HERZOG
3. Automatic COC Flash Point Tester : Model SC335, WALTER HERZOG
4. Automatic Pour Point Tester : Model MP-852 Combi, WALTER HERZOG
5. FTIR Spectrometer : Model FTIR 1650, PERKIN-ELMER
6. Kinematic Viscometer : Model 75943-1, PRECISION
7. NMR Spectrometer : Model DXP-300, Bruker Switzerland
8. Thermogravimetric Analyzer : Model TGA 7, PERKIN-ELMER

3.3 Experimental Procedure

3.3.1 Synthesis of monoesters ^[25]

General procedure for transesterification of rice bran oil with 1-Butanol :

Place 400 g. of rice bran oil, 130 g. of 1-butanol, 60 ml. of toluene and 1 ml. of concentrated sulfuric acid in a 1 litre round-bottomed flask. Dean and Stark apparatus carrying an efficient reflux condenser was equipped at its upper end. The mixture was refluxed by using a magnetic stirrer in an oil bath at temperature, which is the refluxing temperature of mixture, for 2, 3 and 4 hours, respectively. The water was turned off from time to time. The reaction mixture was allowed to cool to room temperature and then poured into water to separate the ester layer. The mixture was neutralized with saturated sodium bicarbonate solution. The mixture was dissolved in diethyl ether then washed with distilled water to remove glycerol. The organic layer was dried with anhydrous sodium sulfate. The mixture was filtered through a Buchner funnel and the filtrate was returned to the flask. The diethyl ether was evaporated and the excess butanol and toluene were distilled under reduced pressure to obtain butyl ester. The yield of butyl ester was calculated from weight of final products and substrates.

Other monoesters with 1-Hexanol , 1-Octanol , 2-Ethyl-1-hexanol and 4-Methyl-2-pentanol were synthesized under the same conditions.

3.3.2 Determination of physical and chemical properties of monoester products

3.3.2.1 Physical properties

Color	by ASTM D 1500
Kinematic viscosity	by ASTM D 445
Viscosity index	by ASTM D 2270
Pour point	by ASTM D 97
Flash point	by ASTM D 92

3.3.2.2 Chemical properties

The characterization of monoester products were determined by ^{13}C -NMR and FTIR analyses.

The oxidation point and the percentages of oxidative compounds were determined by TGA method under air atmosphere and used the following conditions :

TGA conditions :

Heating rate	:	5°C / min.
Temperature range	:	Ambient to 650°C
Atmosphere	:	Dynamic air 20 ml./min.
Reference	:	Al_2O_3

3.3.3 Blending

General procedure for blending monoester with mineral base oil

The quantity of the suitable selected monoester varied from 3 to 25 % was mixed with mineral base oil, 150 SN , in 600 ml. beaker equipped with a mechanical stirrer. The mixture was stirred until homogeneous then transfer to the bottle.

3.3.4 Determination of physical properties from blending product

Color	by ASTM D 1500
Kinematic viscosity	by ASTM D 445
Viscosity index	by ASTM D 2270
Pour point	by ASTM D 97
Flash point	by ASTM D 92