CHAPTER III

3.1 Experimental

3.1.1 Materials used

Methyl methacrylate was purchased from Merck India Ltd. Styrene and 1-decene was purchased from SIGMA-ALDRICH. Co. Toluene was procured from S.D. Fine Chem., India Ltd. and benzoyl peroxide (BZP) from LOBA Chemicals. Base oils were collected from different sources Indian Oil Corporation Ltd., Dhakuria, Kolkata and BPC Ltd., Matigara, Darjeeling.

3.1.2 Purification of the materials

Methyl methacrylate (MMA) was shaken two to three times with 5% sodium hydroxide to eliminate hydroquinone inhibitor, dried over anhydrous CaCl₂ for 7-8 h and distilled. Benzoyl peroxide (BZP) was used as initiator after recrystallization from CHCl₃-MeOH mixture. Toluene was purified by distillation after being refluxed for 2 h in presence of sodium.

3.1.3 Polymerization

The polymerization was carried out in a four necked round bottom flask equipped with a stirrer, condenser, thermometer, an inlet for the introduction of nitrogen and a dropping funnel through which to add styrene drop wise. In the flask was placed desired mass of MMA and initiator(BZP) followed by the desired mass of styrene was added drop wise for 2 h in the presence of toluene as solvent. The reaction temperature was maintained at 353K for 6 h. At the end of the reaction time, the reaction mixture was poured into methanol with stirring to terminate the polymerization and precipitate the polymer. The polymer was further purified by repeated precipitation of its hexane solution by methanol followed by drying under vacuum at 313K. A homo polymer of MMA and copolymer with 1-decene were similarly prepared and purified under the same conditions for use in reference.
3.1.4 Spectroscopic Measurements

IR spectra were recorded on a Shimadzu FT-IR 8300 Spectrometer and on Perkin Elmer FT-IR Spectrometer using 0.1mm KBr cells and the spectra were recorded at room temperature within the wave number range 400 to 4000 cm\(^{-1}\). NMR spectra were recorded in Brucker Avance 300MHz FT-NMR spectrophotometer and Varian 200MHz spectrophotometer using 5mm BBO probe. CDCl\(_3\) was used as solvent and TMS as reference material. Average molecular weights (\(M_n\) and \(M_w\)) were obtained by SEC (GPC) using THF as mobile phase in a Water GPC system at 303 ± 1 K. The retention times were calibrated against known monodisperse polystyrene standards [15].

3.1.5 Viscometric measurements

Viscometric properties were determined at 313 K in toluene solution, using an Ubbelohde OB viscometer. Experimental determination was carried out by counting time flow at least nine different concentration of the sample solutions. The time flow of the solution was manually determined by using a chronometer. In a single measurement the lowest value of solution concentration was chosen for the calculation. The viscometer was calibrated frequently with distilled water. The viscosity results were checked against viscosity of known solutions and uncertainty was found to be nearly 0.2 %. Precautions regarding prevention of evaporation of solvent were taken in all the cases. For the viscosity – average molecular weight determination, the constants \(K = 0.00387\) dl/g (deciliter/g) and \(a = 0.725\) [44, 45] were employed.

3.1.6 Thermo gravimetric analysis (TGA)

The thermograms in air were obtained on a mettler TA - 3000 system, at a heating rate of 10 K.min\(^{-1}\).
3.1.7 Shear Stability

Tests were conducted as per ASTM D-3945 method and kinematic viscosity of fresh toluene and sheared polymer solution in toluene was determined by ASTM D-445 and D-4741 procedure respectively.

3.1.8 Evaluation of prepared polymer as pour point depressant in base oils

The prepared additives were evaluated as pour point depressant using base oils (B1 and B2) each of them collected from two different sources (S1 and S2), through the pour point test according to the ASTM–D-97 method using WIL-471 cloud and pour point test apparatus model 3 (India). The effect of additive concentration was investigated by using different doping concentrations: 0.25%, 0.50%, 1%, 2%, 3%, and 5% (w/w). The experimental data were noted by taking an average of three experimental results under identical conditions.

3.1.9 Evaluation of prepared polymer as viscosity index improvers in base oils

The various blends were prepared by using two different types of base stocks (B1 and B2). Viscosity index of these oils were calculated by ASTM D2270–87 method using viscosity index calculator. The kinematic viscosity of the polymer doped base oils was determined at 313K and 373K. Range of concentration of the additives (in lube oils) used to study the effect of concentration on VI of the lube oil, were 0.25%, 0.50%, 1%, 2%, 3%, and 5% (w/w).

3.1.10 References


[39] Vipper, A. B.; Krien, S. E.; Sher, V. V.; Sanin, P. J.; *Seventh World Petroleum Congress*, 1967, VIII, 73.


