CHAPTER 2

EXPERIMENTAL SECTION

2.1 MATERIALS

p-Hydroxycinnamic acid (Acros), 4-(N,N-dimethylamino)pyridine (DMAP) (Spectrochem), 1,3-dicyclohexylcarbodiimide (DCC) (Spectrochem), 47% HBr (SRL), 1,6-hexanediol, 1,8-octanediol, 1,10-decanediol, anhydrous potassium carbonate, methacrylic acid, phenol and 4-cyanophenol (Merck), 4-methoxyphenol (SRL), 4-hydroxy-4'-methoxybiphenyl, 4-cyano-4'-hydroxybiphenyl, 4-biphenylol (Lancaster) and Azobisisobutyronitrile (AIBN) (Merck) were used without further purification. Methanol, ethanol, dichloromethane, tetrahydrofuran, triethylamine, benzene and toluene (SRL) were purified and dried by the reported procedure (Furniss et al 1994, Perrin and Armarego 1988). Silica gel (60-120 mesh) (SRL) was dried in oven at 110°C for 1 h and cooled in a desiccator before use.

2.2 PURIFICATION OF SOLVENTS

2.2.1 Benzene

Benzene (Merck) (500 mL) was shaken with about 15% of its volume of conc. H₂SO₄ until free from thiophene, then washed with water and 10% sodium carbonate solution, again with water and dried in fused calcium chloride and distilled. The fraction boiling at 80°C was collected and stored over metallic sodium wire (lit. b.p. 80.1°C, Perrin and Armarego 1988).
2.2.2  Toluene

Toluene (Spectrochem) (1000mL) was shaken twice with conc. H$_2$SO$_4$ (100 mL of acid per liter), water, 5% aqueous sodium bicarbonate solution, again with water, then dried over calcium sulphate. Finally distilled over phosphorus pentoxide after refluxing for 30 min (b.p.110°C, Perrin and Armarego 2003).

2.2.3  Dichloromethane

The commercial grade dichloromethane (500 mL) was purified by washing with portion of conc. H$_2$SO$_4$ until the acid layer remains colorless and washed with 5% aqueous sodium carbonate solution then with distilled water. It was dried initially over calcium chloride and then distilled from phosphorus pentoxide before use. The fraction boiling at 40°C was collected (lit. b.p.40°C, Perrin and Armarego 1988).

2.2.4  Tetrahydrofuran (THF)

Tetrahydrofuran (SRL) (1000mL) was shaken with sodium hydroxide pellets and distilled. The distillate was refluxed over anhydrous calcium sulphate, filtered and refluxed for 6 h on a water bath and distilled. The fraction boiling at 65°C was collected and stored over metallic sodium wire (lit. b.p. 65-67°C, Furniss et al 1994).

2.2.5  Ethanol

Rectified spirit (1000 mL) was refluxed with calcium oxide for 6h, allowed to stand overnight and distilled. The fraction distilling at 80°C was collected (lit.b.p.80°C) (Furniss et al 1994).
2.2.6 Methanol

Dry methanol was obtained by refluxing the commercial methanol (SRL) (1000 mL) over anhydrous calcium oxide and distilled. The distilled methanol was treated with magnesium metal and re-distilled. The fraction boiling at 65°C was collected (lit. b.p. 65°C, Furniss et al 1994).

2.2.7 Thionyl chloride

Thionyl chloride (SD Fine) (500 mL) was distilled using Bunsen burner. The fraction distilled at 77°C was collected. The rate of heating was controlled intermittently for smooth distillation (lit. b.p. 76-78°C, Furniss et al 1994).

2.2.8 Water

Water (1 L) was distilled with 10 g of potassium permanganate and sodium hydroxide. The distilled water was collected and then re-distilled to get double distilled water (b.p. 100 °C, Furniss et al 1994).

2.3 PURIFICATION OF REAGENTS
2.3.1 Triethylamine (TEA)

Triethylamine was dried over potassium hydroxide and distilled. The distillate was re-distilled from sodium wire. The fraction boiling at 89°C was collected (lit. b.p. 89.4°C, Perrin and Armarego 1988).

2.3.2 Methacryloyl chloride

A mixture of methacrylic acid (0.5 mol), benzoyl chloride (1.5 mol) and hydroquinone (0.001 mol) were taken in a 1000 ml round bottom flask and distilled at a fairly rapid rate. The fraction distilling between 120-130°C
at 760 mm Hg was collected which was redistilled in the presence of hydroquinone. The fraction boiling around 95-97°C/740 mm Hg was collected. Yield 65% (b.p. 97°C/740 mm Hg).

2.3.3  Phenol

Steam was passed through a boiling solution containing phenol (1 mol) and sodium hydroxide (1.2-2.0 mol) in 5 L of water until non-acidic material had distilled. The residue was cooled, acidified with H₂SO₄ (20% v/v), and the phenol was separated, dried with calcium sulphate and fractionally distilled under reduced pressure. Fraction boiling at 85°C was collected (lit. b.p. 85-86°C/20 mm Hg, Perrin and Armarego 2003).

2.4  SYNTHESIS OF PRECURSORS

2.4.1  Synthesis of ω-Bromo-1-alkanols

ω-Bromo-1-alkanols namely, 6-bromo-1-hexanol, 8-bromo-1-octanol and 10-bromo-1-decanol were prepared by reacting the corresponding α,ω-alkane diols with 47% HBr. A typical procedure employed for the synthesis is as follows:

2.4.1.1  6-Bromo-1-hexanol

1,6-Hexanediol (0.1mol) and 47% HBr (0.05 mol) solution in 200 mL benzene were distilled azeotropically until the water was completely removed. The benzene layer was washed twice with 200mL of water for the
removal of unreacted HBr and 1,6-hexanediol, 200mL of 6N sodium hydroxide solution followed by 200 mL of 10% HCl solution and finally with 300 mL of saturated brine solution. After drying the organic layer over anhydrous sodium sulphate for 24h, benzene was removed by vacuum distillation to yield a crude brown colored oil. Vacuum distillation of the crude product gave 0.08 mol of 6-bromo-1-hexanol as a colorless oil (b.p.120°C/2 mm Hg), Yield 61% (Goldsmith et al 1975).

2.4.1.2 8-Bromo-1-octanol and 10-bromo-1-decanol

1,8-Octanediol (0.1 mol) was dissolved in 1000 mL of toluene in a two neck 2 L round bottomed flask, the solution was refluxed for 1 h and then 47% HBr (0.05 mol) was added drop wise to the reaction mixture for the period of 2 h. The above reaction mixture was refluxed until the complete removal of water. Then the solution was kept at 0°C for overnight. The unreacted 1,8-octanediol crystallizes out, was filtered. The filtrate was washed twice with 200 mL of water for the removal of unreacted HBr, 100 mL of 6 N sodium hydroxide solution, followed by 100 mL of 10% HCl solution and finally with 150 mL of saturated brine solution. After drying the organic layer with anhydrous sodium sulphate, toluene was removed by vacuum distillation. Distillation of the crude product under reduced pressure gave 8-bromo-1-octanol (Yield 70%). The similar procedure was adopted for the preparation of 10-bromo-1-decanol (Yield 69.8%).
2.4.2 Synthesis of 4-(m-hydroxyalkyloxy)cinnamic acids (m-HACA) 
(m = 6,8,10)

4-(m-Hydroxyalkyloxy)cinnamic acids namely 4-(6-hydroxy hexyloxy)cinnamic acid, 4-(8-hydroxyoctyloxy)cinnamic acid and 4-(10-hydroxydecyloxy)cinnamic acid were prepared by reacting the corresponding o-bromo-1-alkanols with 4-hydroxycinnamic acid. A typical procedure employed for the synthesis is as follows:

2.4.2.1 Synthesis of 4-(6-hydroxyhexyloxy)cinnamic acid (HHCA)

A solution of potassium hydroxide (0.11 mol) and water (20 mL) were added dropwise to a solution of 4-hydroxycinnamic acid (0.046 mol) in ethanol (500 mL). 6-Bromo-1-hexanol (0.055 mol) was then added dropwise to this solution followed by potassium iodide (0.00125 mol) in one portion. The reaction mixture was refluxed overnight, then poured into water (500 mL) and neutralized with 10% hydrochloric acid (20 mL) solution. The resultant precipitate was filtered off. The solid residue was then recrystallized from ethanol to yield the desired product (Yield 65%) (Han et al 1992 and 2000).
2.4.2.2 Synthesis of 4-(8-hydroxyoctyloxy)cinnamic acid (HOCA) and 4-(10-hydroxydecyloxy)cinnamic acid (HDCA)

A solution of potassium hydroxide (0.11 mol) and water (20 mL) were added dropwise to a solution of 4-hydroxycinnamic acid (0.046 mol) in ethanol (500 mL). 8-Bromo-1-octanol (0.055 mol) was then added dropwise to this solution followed by potassium iodide (0.00125 mol) in one portion. The reaction mixture was refluxed overnight, then poured into water (100 mL) and neutralized with 10% hydrochloric acid (20 mL) solution. The resultant precipitate was filtered off. The solid residue was then recrystallized from ethanol to yield the desired product (Yield 65%).

The similar procedure was adopted for the preparation of 4-(10-hydroxydecyloxy)cinnamic acid (Yield 67%).

2.4.2.3 Synthesis of 4-(6-methacryloyloxyhexyloxy)cinnamic acid (MHCA)

4-(6-Hydroxyhexyloxy)cinnamic acid (0.030 mol) taken in a 250 mL round bottomed flask was dissolved in THF (25 mL) then TEA (0.02 mol) was added and allowed to stir for half an hour. The mixture was cooled to 5°C, and methacryloyl chloride (0.031 mol) was added dropwise. This mixture was allowed to stir overnight. The precipitated triethylamine hydrochloride was filtered and the filtrate evaporated under reduced pressure
keeping the bath temperature below 40°C. The residue obtained was treated with petroleum ether to remove unreacted methacryloyl chloride. The precipitate thus obtained was recrystallized using ethanol (Yield 72%).

2.4.2.4 Synthesis of 4-(8-methacryloyloxyoctyloxy)cinnamic acid (MOCA) and 4-(10-methacryloyloxydecyloxy)cinnamic acid (MDCA)

![Chemical Structure of MOCA and MDCA](image)

4-(8-Hydroxyoctyloxy)cinnamic acid (0.030 mol), taken in a 250 mL round bottomed flask was dissolved in THF (25 mL), then TEA (0.02 mol) was added and allowed to stir for 30 min. The mixture was cooled to 5°C and methacryloyl chloride (0.031 mol) was added dropwise. This mixture was allowed to stir overnight. The precipitated triethylamine hydrochloride was filtered and filtrate was evaporated under reduced pressure keeping the bath temperature below 40°C. The residue obtained was treated with petroleum ether to remove the unreacted methacryloyl chloride. The precipitate thus obtained was recrystallized using ethanol (Yield 71%).

The similar procedure was adopted for the preparation of 4-(10-methacryloyloxydecyloxy)cinnamic acid (Yield 69%).

2.5 SYNTHESIS OF MONOMERS

2.5.1 Synthesis of Phenyl-4-(m-methacryloyloxyalkyloxy)cinnamate (m=6,8,10) (ia, id and ig)

Phenyl-4-(m-methacryloyloxyalkyloxy)cinnamate namely phenyl-4-(6-methacryloyloxyhexyloxy)cinnamate, phenyl-4-(8-methacryloyloxyocty
loxy)cinnamate and phenyl-4-(10-methacryloyloxydecyloxy)cinnamate were prepared by reacting the corresponding 4-(m-methacryloyloxyalkyloxy)cinnamic acids with phenol. A typical procedure employed for the synthesis of monomers is as follows:

2.5.1.1 Synthesis of phenyl-4-(6-methacryloyloxyhexyloxy)cinnamate (ia)

A mixture of 4-(6-methacryloyloxyhexyloxy)cinnamic acid (0.0126 mol), phenol (0.0130 mol), 4-(N,N-dimethylamino)pyridine (0.001 mol) and 1,3-dicyclohexylcarbodiimide (DCC) (0.146 mol) were added into 100 mL of dry methylene chloride and stirred at room temperature for overnight. The precipitated urea was removed by filtration and the solution was washed with 5% acetic acid followed by saturated brine solution. Organic layer was dried over anhydrous sodium sulphate solution and removed under vacuum distillation. The residue obtained was purified by column chromatography using 5% methanol in chloroform as eluent (Yield 66%).

All other monomers (id and ig) were prepared by adopting the similar procedure using respective 4-(m-methacryloyloxyalkyloxy)cinnamic acids(m-8,10) with phenol.
2.5.2 Synthesis of 4-methoxyphenyl-4′-(m-methacryloyloxyalkyloxy) cinnamates (m=6,8,10) (ib, ie and ih)

4-Methoxyphenyl-4′-(m-methacryloyloxyalkyloxy)cinnamates namely, 4-methoxyphenyl-4′-(6-methacryloyloxyhexyloxy)cinnamate, 4-methoxyphenyl-4′-(8-methacryloyloxyoctyloxy)cinnamate and 4-methoxyphenyl-4′-(10-methacryloyloxydecyloxy)cinnamate were prepared by reacting the corresponding 4-(m-methacryloyloxyalkyloxy)cinnamic acid with 4-methoxyphenol. A typical procedure employed for the synthesis of the monomers is as follows:

2.5.2.1 Synthesis of 4-methoxyphenyl-4′-(6-methacryloyloxyhexyloxy) cinnamate (ib)

A similar procedure followed for the synthesis of ia was adopted for the preparation of 4-methoxyphenyl-4′-(6-methacryloyloxyhexyloxy) cinnamate.

All other monomers (ie and ih) were prepared by adopting the similar procedure using respective 4-(m-methacryloyloxyalkyloxy)cinnamic acids (m-8,10) with 4-methoxyphenol.
2.5.3 Synthesis of 4-cyanophenyl-4’-(m-methacryloyloxyalkyloxy) cinnamate (m=6,8,10) (ic, if and ii)

4-Cyanophenyl-4’-(m-methacryloyloxyalkyloxy)cinnamates namely, 4-cyanophenyl-4’-(6-methacryloyloxyhexyloxy)cinnamate, 4-cyanophenyl-4’-(8-methacryloyloxyoctyloxy)cinnamate and 4-cyanophenyl-4’-(10-methacryloyloxydecyloxy)cinnamate were prepared by reacting the corresponding 4-(m-methacryloyloxyalkyloxy)cinnamic acids with 4-cyanophenol. A typical procedure employed for the synthesis of the monomers is as follows:

2.5.3.1 Synthesis of 4-cyanophenyl-4’-(6-methacryloyloxyhexyloxy) cinnamate (ic)

The procedure followed for the synthesis of ia was adopted for the preparation of 4-cyanophenyl-4’-(6-methacryloyloxyhexyloxy)cinnamate.

All other monomers (if and ii) were prepared by adopting the similar procedure using respective 4-(m-methacryloyloxyalkyloxy)cinnamic acids (m=8,10) with 4-cyanophenol.
2.5.4 Synthesis of 4-biphenyl-4'- (m-methacryloyloxyalkyloxy) -cinnamate (m=6,8,10) (iia, iid and iig)

4-Biphenyl-4’-(m-methacryloyloxyalkyloxy)cinnamates namely, 4-biphenyl-4’-(6-methacryloyloxyhexyloxy)cinnamate, 4-biphenyl-4’-(8-methacryloyloxyoctyloxy)cinnamate and 4-biphenyl-4’-(10-methacryloyloxydecyloxy)cinnamate were prepared by reacting the corresponding 4-(m-methacryloyloxyalkyloxy)cinnamic acids with 4-biphenylo1. A typical procedure employed for the synthesis of the monomers is as follows:

2.5.4.1 Synthesis of 4-biphenyl-4’- (6-methacryloyloxyhexyloxy) cinnamate (iia)

A mixture of 4-(6-methacroyloxyhexyloxy)cinnamic acid (0.0126 mol), 4-biphenylo1 (0.0129 mol), 4-(N,N-dimethylamino)pyridine (0.001 mol) and N,N-dicyclohexylcarbdodiimide (DCC) (0.0146 mol) were added into 100 mL of dry methylene chloride and stirred at room temperature for overnight. The precipitated urea was removed by filtration and the solution was washed with 5% acetic acid followed by saturated brine solution. Organic layer was dried over anhydrous sodium sulphate and removed under vacuum distillation. The residue obtained was purified by column chromatography using 5% methanol in chloroform as eluent (Yield 68%).
All other monomers iid and iig were prepared by adopting the similar procedure using respective 4-(m-methacryloyloxyalkyloxy) cinnamic acids (m-8,10) with 4-biphenylol.

2.5.5 Synthesis of 4-(4’-methoxybiphenyl)yl-4”-(m-methacryloyloxyalkyloxy)cinnamate (m=6,8,10)

4-(4’-methoxybiphenyl)yl-4”-(m-methacryloyloxyalkyloxy) cinnamates namely, 4-(4’-methoxybiphenyl)yl-4”-(6-methacryloyloxyhexyloxy)cinnamate, 4-(4’-methoxybiphenyl)yl-4”-(8-methacryloyloxyoctyloxy)cinnamate and 4-(4’-methoxybiphenyl)yl-4”-(10-methacryloyloxydecyloxy)cinnamate were prepared by reacting the corresponding 4-(m-methacryloyloxyalkyloxy) cinnamic acid with 4-hydroxy-4’-methoxybiphenyl. A typical procedure employed for the synthesis of the monomers is as follows:

2.5.5.1 Synthesis of 4-(4’-methoxybiphenyl)yl-4”-(6-methacryloyloxyhexyloxy)cinnamate (iib)

The procedure followed for the synthesis of iia was adopted for the preparation of 4-(4’-methoxybiphenyl)yl-4”-(6-methacryloyloxyhexyloxy) cinnamate.
All other monomers (ii e and ii h) were prepared by adopting the similar procedure using respective 4-(m-methacryloyloxyalkyloxy)cinnamic acids (m=8,10) with 4-hydroxy-4’-methoxybiphenyl.

2.5.6 Synthesis of 4-(4’-cyanobiphenyl)yl-4''-(m-methacryloyloxyalkyloxy)cinnamate (m=6,8,10) (iic, iif and iii)

4-(4’-Cyanobiphenyl)yl-4’’-(m-methacryloyloxyalkyloxy) cinnamates namely, 4-(4’-cyanobiphenyl)yl-4’’-(6-methacryloyloxyhexyloxy)cinnamate, 4-(4’-cyanobiphenyl)yl-4’’-(8-methacryloyloxyoctyloxy)cinnamate and 4-(4’-cyanobiphenyl)yl-4’’-(10-methacryloyloxydecyloxy)cinnamate were prepared by reacting the corresponding 4-(m-methacryloyloxyalkyloxy) cinnamic acids with 4-cyano-4’-hydroxybiphenylol. A typical procedure employed for the synthesis of the monomers is as follows:

2.5.6.1 Synthesis of 4-(4’-cyanobiphenyl)yl-4’’-(6-methacryloyloxyhexyloxy)cinnamate (iic)

The procedure followed for the synthesis of iia was adopted for the preparation of 4-(4’-cyanobiphenyl)yl-4’’-(6-methacryloyloxyhexyloxy)cinnamate.
All other monomers (iif and iii) were prepared by adopting the similar procedure using respective 4-(m-methacryloyloxyalkyloxy)cinnamic acids (m-8,10) with 4-cyano-4’-hydroxybiphenyl.

2.6 POLYMERIZATION

All the polymers Ia – IIi were synthesized by a free radical solution polymerization from corresponding monomers using AIBN as initiator in THF at 60°C. A typical procedure for the synthesis of poly[4-x-phenyl-4’-(m-methacryloyloxyalkyloxy)cinnamate]s and poly[4-(4’-x-biphenyl)y1-4′′-(m-methacryloyloxyalkyloxy)cinnamate]s is as follows:

2.6.1 Synthesis of Poly [4-x-phenyl-4’-(m-methacryloyloxyalkyloxy) cinnamate]s (Ia – II)

The typical procedure for the synthesis of polymer Ia is as follows: phenyl-4-(6-methacryloyloxyhexyloxy)cinnamate (0.01 mol) and AIBN (0.001 mol) were dissolved in dry THF and a gentle steam of nitrogen was passed into the solution. The solution was kept in an oil bath at 60°C for 24 h.
Then the solution was cooled and poured into excess of methanol to precipitate the polymer. The crude polymer thus obtained was reprecipitated twice using chloroform and methanol. The purified polymer was dried at 25°C under vacuum for 48 h (Yield 75%). All other polymers (Ib – Ii) were prepared by adopting the similar procedure using respective monomers.

2.6.2 Synthesis of Poly[4-(4’-x-biphenyl)yl-4″-(m-methacryloyloxy alkyloxy)cinnamate]s (IIa – IIIi)

The typical procedure for the synthesis of polymer IIa is as follows: 4-biphenylyl-4’-(6-methacryloyloxyhexyloxy)cinnamate (0.01 mol) and AIBN (0.001 mol) were dissolved in dry THF and a gentle steam of nitrogen was passed into the solution. The solution was kept in an oil bath at 60°C for 24 h. Then the solution was cooled and poured into excess of methanol to precipitate the polymer. The crude polymer thus obtained was reprecipitated twice using chloroform and methanol. The purified polymer was dried at 25°C.
under vacuum for 48 h (Yield 77%). All other polymers (IIb–IIi) were prepared by adopting the similar procedure using respective monomers.

2.7 CHARACTERIZATION OF POLYMERS

2.7.1 Solubility test

Solubility of the polymers was tested in various solvents viz., benzene, chloroform, dichloromethane, DMF and ethanol. 2-3 mg of the polymer was treated with 5 mL of solvent and set aside for 6 h with occasional shaking. If the polymer was insoluble in cold condition, the mixture heated and cooled.

2.7.2 Viscosity Measurement

A polymer solution of 0.5 g dL\(^{-1}\) concentrations was prepared in dichloromethane solvent. The polymer solution was taken in an Ubbelohde suspended level viscometer and placed in a thermostat at 30°C ± 2. The flow time for the polymer solution (t) and solvent (t\(_0\)) were noticed at same temperature. Inherent viscosities of the polymer solutions were determined using the following expression;

Relative viscosity \( (\eta_r) = \frac{\eta}{\eta_o} = \frac{t}{t_0} \)

Inherent viscosity \( (\eta_{inh}) = \frac{(\ln \eta_r)}{C} \)

where \( \eta \) and \( \eta_o \) are the viscosities of the polymer solution and the solvent respectively.
2.7.3 FT-IR Spectroscopy

Thermomattson Satellite Model FTIR spectrometer was used to substantiate the formation of products in this study. The spectra recorded for liquid samples were made into a thin film in between two KBr windows. On the other hand, solid samples were recorded by making potassium bromide (Merck, IR Grade) pellets. All the spectra were recorded with a maximum of 100 scans. A background spectrum was run before recording the spectra for each sample. The spectral calibration of the instrument was made using a polystyrene film at regular intervals of time.

2.7.4 NMR Spectroscopy

High-resolution $^1$H and $^{13}$C-NMR spectra were recorded using Brucker MSL 300P, 300 MHz NMR spectrometer. Deutrated chloroform [Aldrich, CDCl$_3$, 99.8% containing 0.03%v/v tetramethylsilane (TMS)] was used as solvents for recording NMR spectra. The proton NMR spectra were recorded using broadband inverse probe where the inner coil for the protons and outer coil for ‘X’ nuclei. Solvent suppression was applied in some cases where the solvent signal is very strong compared to the sample signals. $^{13}$C spectra were recorded in the dual ($^{13}$C/$^1$H) probe where the inner coil for $^{13}$C and the outer coil for protons.

2.7.5 Thermogravimetric Analysis (TGA)

Thermogravimetric analysis was made using NETZSCH - Gerätebau GmbH NETZSCH - Gerätebau GmbH Thermal Analysis system. All the synthesized polymers were scanned in the DTA/TG Thermal Analysis system. All the runs were carried out under nitrogen atmosphere with gas flow rate of 10ml/min. All the experiments were carried out at a
heating rate of 10°C/min unless otherwise specified. α-Alumina was used as the difference on platinum pans. The thermal analyzer was calibrated using calcium sulphate as standard.

2.7.6 **Differential Scanning Calorimetry (DSC)**

Differential scanning calorimetry was performed using NETZSCH - Gerätebau GmbH Thermal Analysis system. All the synthesized polymers were scanned in the DTA/TG thermal analysis unit attached to a DSC 220°C module. The experiments were carried out in nitrogen atmosphere at a heating rate of 5°C/min from ambient to 400°C with a nitrogen flow of 10 mL/min. All the samples were crimped in aluminium pans to ensure good thermal contact between the sample and the pan. The instrument was calibrated for enthalpy and the temperature values using pure tin and indium. Controlled cooling measurements were performed with liquid nitrogen using an automatic cooling accessory attached to the DSC 220°C module.

2.7.7 **Polarizing Optical Microscope**

Hot stage optical polarizing microscopic analysis was investigated on a Euromax optical polarizing microscope coupled with Linkam HFS 91 heating stage and a TP-93 programmer. Small quantities of the powdered polymer was placed between two corning glass plates and mounted on heating stage, which is placed over optical microscope. The sample was heated at a slow rate (5°C/min) with continuous monitoring of the sample. The photographs were taken using Nikon (FM10) camera loaded with Kodak 200 film and were taken at 20X magnification.
2.7.8 UV Spectroscopy

The photocrosslinking ability of the polymers were investigated in the form of dilute solutions and absorption between 250-400 nm, on a Shimadzu UV-160A UV-Visible spectrophotometer. A typical procedure is as follows: A solution was poured into a 1 cm quartz cuvette. The photochemical studies were carried out in a discontinued mode i.e. the sample was exposed to UV irradiation from a 125W medium pressure mercury lamp kept at a distance of 10 cm from the sample at various intervals of time and immediately the UV absorption of the medium was measured on the spectrophotometer. This procedure was repeated until reduction in absorption was completed.