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Synthesis, Characterizations, and Liquid Crystalline Property of Photoresponsive Poly (2-[4-Methoxy Benzoyl Styryloyloxy] Ethyl Methacrylate)

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A novel liquid crystalline mesophase as well as photoreactive property of photo-cross-linkable liquid crystalline polymer has been studied in detail. The crosslinkable liquid crystalline polymer was synthesized by newer route and its structure has been characterized by ^{1}H and ^{13}C-Nuclear Magnetic Resonance, Fourier Transformation Infrared, and ultraviolet spectral studies. The photocrosslinking property was studied by using both ultraviolet and fluorescence spectral techniques. The thermogravimetric analysis was used to determine the thermal stability of the polymer. Molecular weight of the polymer was determined using gel permeation chromatography technique. The grainy texture mesophase of the polymer was identified by Differential Scanning Calorimeter thermogram and also from the hot stage optical polarization microphotograph images, and all the results obtained were discussed. The morphology of the polymer network was observed using scanning electron microscopy before and after photocrosslinking process.

Keywords Differential scanning calorimetry (DSC); hot stage optical polarized microscopy (HOPM); irradiation effects; liquid crystals; thermal properties

1. Introduction

A side chain liquid crystalline polymer (SC-LCP) comprises a polymer backbone, a mesogenic unit, and a flexible spacer in which the mesogenic unit and the polymer backbone are bridged together. Based on mesogen attached to the polymer backbones, SC-LCPs can be categorized into either "side-end-fixed" or "side-on-fixed" type [1,2]. Liquid crystalline polymers have generated considerable interest in recent years and the photo-cross-linkable LCPs have driven special attention if they contain both mesogen and photoactive groups in their structure [3–7]. Mesogens incorporate liquid crystalline (LC) properties to the polymer and photoreactive group facilitates cross-linking of the chain under the influence of UV light radiation. These types of polymers were useful in fabricating anisotropic networks and thin films, information storage devices [8], non-linear optical (NLO) devices [9,10] aligned membrane for permeation of gases and drugs, etc. [11]. The liquid crystalline property of polymers depends on the nature of the polymer backbone, type of the mesogen, the flexible spacer and its length, and nature of the terminal groups [12–16]. Among many promising photo-cross-linkable groups, the chalcone group has been well

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recognized and can be incorporated in photo-cross-linkable side chain polymers. The effect of photocrosslinked chalcone moiety on the stability of photochromism was investigated intensively [17–19]. A chemically different approach to convert a soluble material into an insoluble material can be achieved by using photocrosslinking reaction. This technique has been well established in the coatings industry and in photoresist technology. The resulting crosslinked polymer layers are insoluble and not meltable [20]. The polymers that carry dual properties of both liquid crystalline and photocrosslinking seem to be indispensable material in NLO applications [21].

The present paper reports a hitherto unreported newer route of Photoresponsive LCP containing chalcone moiety, which is responsible for photocrosslinking without photosensitizer in the presence of UV irradiation.

2. Experimental

2.1. Materials

4-Formyl benzoic acid (FBA), methoxy acetophenone, and benzoylperoxide (BPO) were obtained from Sisco Research Laboratories (SRL) and 2-hydroxy ethyl methacrylate (HEMA) was obtained from Fluka (Switzerland) and used without further purification. For making thin layer chromatography (TLC), silica gel-coated aluminum sheet from Merck was used.

2.2. Instruments

The Furier Transformation Infrared (FTIR) spectrum of polymer was recorded on Perkin Elmer FT-IR Spectrometer RXI. The specimen was prepared in the pellet form using KBr. ^{1}H-Nuclear Magnetic Resonance (NMR) spectroscopic measurement was recorded with Bruker MSC 300 spectrometer. Thermal stability of polymer was investigated by thermogravimetric analysis using NETZSCH STA 409 C/CD. The number average molecular weight (M_n) and weight average molecular weight (M_w) of the polymer were determined by PL-GPC 650. Glass transition temperature of polymer was measured from differential scanning calorimeter (DSC) NETZSCH DSC 204. The photocrosslinking studies have been done by Perkin Elmer Lambda 35 UV-visible spectrometer. The fluorescence spectrum of the polymer has been recorded in FluroMax 2.0. The texture of the prepared sample was studied by Euromax polarizing microscope equipped with a Linken HFS91 heating stage. The sample was prepared by a small quantity of the material being melted between two thin glass cover slips to get uniform film and anisotropic behavior observed by heating and cooling with Kodak film.

2.3. Synthesis

2.3.1. 4-methoxy phenyl-4-carboxylic styryl ketone (MPCSK). Methoxy acetophenone 6 g (0.04 mol) and 4-formyl benzoic acid 6 g (0.04 mol) were dissolved in 50 mL of ethanol. A solution of NaOH was prepared by adding 1.6 g of NaOH in 20 mL of distilled water. The NaOH solution was added drop wise to the above mixture with constant stirring at room temperature (27°C) and stirred over night. The reaction mixture was poured into ice water and neutralized with 2 M HCl to form precipitate, which was then filtered and washed with distilled water several times. The crude product was recrystallized from ethanol to obtain yellow crystalline product (**1**) and it was dried under vacuum at 50°C (yield: 70%–75%). The structure of MPCSK was identified by elemental analysis, IR, and proton NMR techniques.

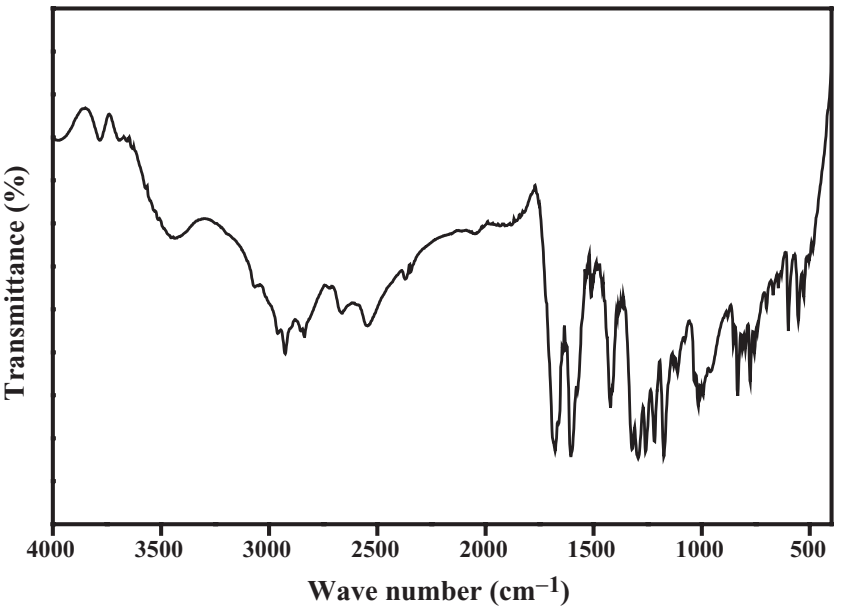


Figure 1. FT-IR spectrum of 4-methoxy phenyl-4carboxylic styryl ketone (MPCSK).

Elemental Analysis Calculated for $C_{17}H_{14}O_4$: C, 72.33%; H, 4.99%. Found: C, 72.21%; H, 4.94%; IR (KBr, cm^{-1}) (Fig. 1): 1511, 1535 (aromatic ring); 1605 and 1678 (olefinic −CH=CH−); 3437 (OH stretching of COOH); 1678 (>C=O); 2837 (C−H out of plane bending): ^{1}H-NMR ($CDCl_3$, TMS) (Fig. 2): 10.1 (acidic −OH); 6.8–8.2 (8H, aromatic); 5.8 and 6.1 (2H, −CH=CH−); 4.5(3H, $-OCH_3$).

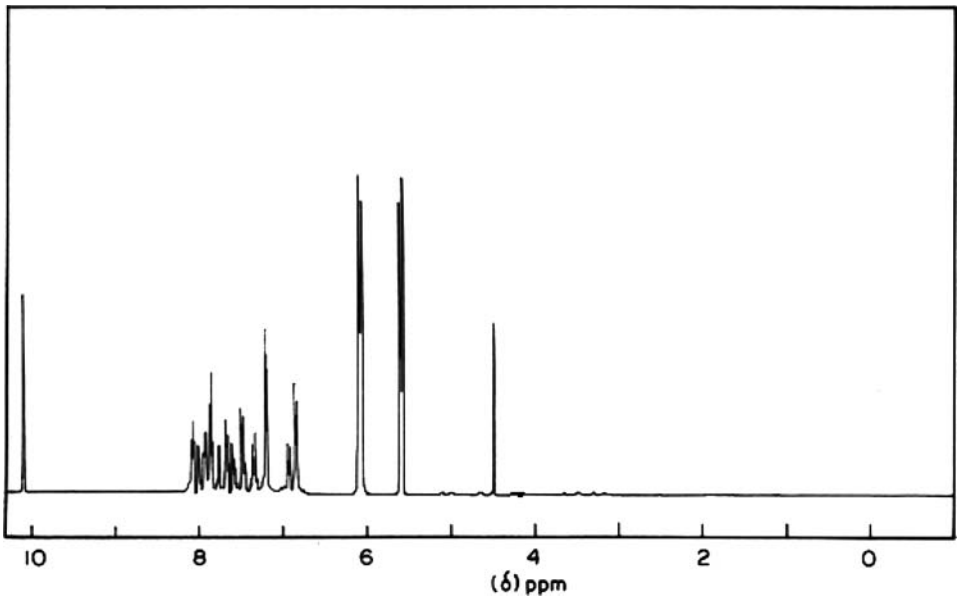


Figure 2. NMR-spectrum of 4-methoxy phenyl-4carboxylic styryl ketone (MPCSK).

2.3.4. 2-[4-methoxy benzoyl styryloyloxy] ethyl methacrylate (MBSOEMA). In 250-mL two-neck flask, predetermined volume of triethyl amine and 2-hydroxy ethyl methacrylate were dissolved in tetrahydrofuran (THF) and was allowed to cool between −5°C and 0°C. The chlorination of MPCSK was carried out as described by Kadokawa et al. [22] to get the intermediate acid chloride compound **2**. After chlorination of MPCSK, benzene was removed by distillation at 100°C. The intermediate acid chloride compound **2** was dissolved in THF, then added dropwise for 30 min to the mixture of triethyl amine and 2-hydroxy ethyl methacrylate maintained between −5°C and 0°C with constant stirring. The above reaction mixture was allowed to stir for overnight and quaternary ammonium salt was filtered. THF was removed by rotary evaporation; the crude monomer was purified by column chromatography using 96:4 percentage ratio of hexane and ethylacetate mixture. The product obtained was dark brown solid of 2-[4-methoxy benzoyl styryloyloxy] ethyl methacrylate (MBSOEMA) **3** (yield: 49%–50%). The structure of monomer MBSOEMA was confirmed by elemental analysis, IR, and proton NMR techniques.

Elemental Analysis Calculated for $C_{23}H_{22}O_6$: C, 70.03%; H, 5.62%. Found: C, 69.98%; H, 5. IR (KBr, cm^{-1}) 1690, 1640 (>C=O); 1600 (olefinic, CH=CH); 1745 (ester, >C=O); 2960, 2850 (aliphatic, C−H); 1381, 1450 (−CH_3 bending); 1285, 1165 (C−O stretching); 807, 804, 700 (C−H, out of plane bending); ^{1}H-NMR ($CDCl_3$, TMS): 8–6.8 (8H, aromatic); 5.5, 6.2 (2H, −CH=CH−); 1.5 (2H, methylenic proton) 1.3 (3H, −CH_3).

2.4. Polymerization

2.4.1. Poly (2-[4-methoxy benzoyl styryloyloxy] ethyl methacrylate). The predetermined quantities of MBSOEMA **3**, the initiator (5 wt% of monomer), and the solvent THF were placed in a polymerization tube and the mixture was flushed with a slow stream of nitrogen for 15 min. Then the tube was closed and placed in an oil bath at 70°C for 48 h, subsequently, the contents were added to excess methanol to precipitate the polymer. The crude polymer was purified by redissolving in chloroform and reprecipitated by methanol, filtered, washed with methanol, and dried under vacuum at 50°C (yield: 55%–60%).

2.4.2. Photoreactive Measurements. The photoreactivity of the prepared polymer was studied by dissolving the sample in chloroform and irradiated with UV light at 254 nm using photoreactor kept at a distance of 10 cm from the light source for different time intervals. After each irradiation period, the UV spectra were recorded using Perkin Elmer Scanning spectrometer. The rate of disappearance of double bond in photosensitive group was calculated by the following expression:

$$\text{Rate of conversion}(\%) = (A_0 - A_T/A_0 - A_\infty) \times 100,$$

where A_0, A_T, and A_∞ are absorption intensities due to the >C=C< group after the irradiation time t = 0, t = T, and t = ∞ (maximum irradiation time), respectively.

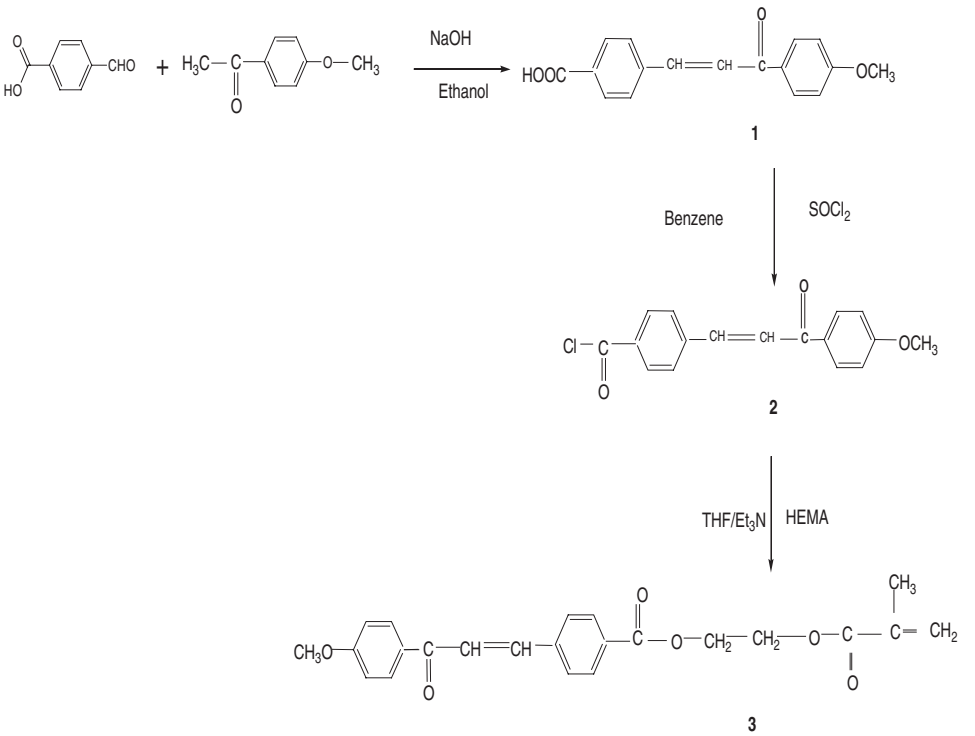
2.4.3. Liquid Crystalline Characterization of Monomeric and Polymeric Liquid Crystals. The liquid crystalline property of both monomer 2-[4-methoxy benzoyl styryloyloxy] ethyl methacrylate and poly (2-[4-methoxy benzoyl styryloyloxy] ethyl methacrylate) (polymer **4**) were characterized by DSC NETZSCH DSC 204 and polarizing microscope equipped with Linken HFS91 heating stage. The DSC thermogram shows mesomorphic transitions of monomer and polymer. It was inferred from the DSC thermogram that two endothermic transitions were observed on heating the sample that revealed the transition at lower

temperature due to melting temperature (T_m) at 75°C and higher temperature at 174°C for grainy mesophase to isotropic transition (T_i). The ethyl methacrylate present on the terminal position of the monomer plays an important role in transition temperature. Polymer **4** showed melting temperature (T_m) at 80°C and isotropic transition (T_i) at 208°C. The T_m and T_i values of polymer were higher than the monomer. HOPM photographs of both monomer and polymer are shown in Figs. 12(a–d). The monomer shows T_m at 73°C (Fig. 12(a)) and isotropic (T_i) from grainy mesophase occurs at 171°C (Fig. 12(b)), whereas polymer showed T_m at 84°C and isotropic from grainy-like mesophase was observed at 210°C.

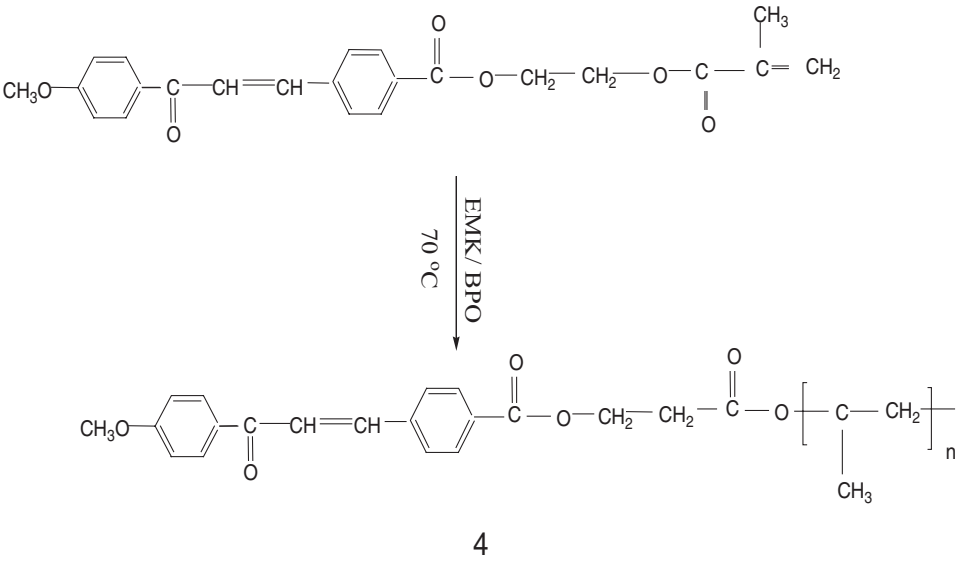
3. Results and Discussion

3.1. Synthesis

The photoresponsive liquid monomer and polymer were prepared by attaching ethylene spacer with photo-cross-linkable chalcone moiety as outlined in Schemes 1 and 2. The mesogenic and photoreactive chalcone moiety **1** was prepared by reacting 4-formyl benzoic acid with methoxy acetophenone in ethanol in the presence of NaOH as base. The ethylene spacer group was attached with chalcone moiety **1** by reacting 2-hydroxy ethyl methacrylate (HEMA) in THF in the presence of triethyl amine from −5°C to 0°C to obtain monomer **3**. The photoresponsive LCP **4** was synthesized by a mixture of predetermined amount of monomer with BPO (5% mol ratio of monomer) in THF at 70°C. The polymerization



Scheme 1. Synthesis of monomer MBSOEMA.



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Scheme 2. Polymerization of MBSOEMA.

was carried out for 48 h. The above contents were poured into methanol and filtered. The structure of LCP was confirmed by FT-IR, ^{1}H-NMR, and ^{13}C-NMR spectroscopy. The weight and number average weight of polymer were determined by the gel permeation chromatography (GPC) technique.

3.2. Solubility Studies

The solubility of the prepared polymer was tested in various solvents. It was soluble in polar aprotic solvents such as dimethyl formamide, dimethyl sulphoxide, N-methylpyrrolidiene, dioxane, THF, and chlorinated solvents such as chloroform, dichloromethane, and chlorobenzene. It was insoluble in methanol, ethanol, 2-propanol, and hydrocarbons such as toluene, benzene, and n-hexane.

3.3. FT-IR Spectrum of Polymer

Figure 3 shows the FT-IR spectrum of polymer **4**. The absorption bands at 2930 cm^{-1} and 1422 cm^{-1} are due to $-CH_2-CH_2-$ group. The aromatic $>C=C<$ stretching was observed at 1510 cm^{-1}. The olefinic double bond shows characteristic bands at 1605 cm^{-1}. The methoxy group shows absorption stretching at 2930 cm^{-1}. The $-C-H$ out of plane bending of aromatic nuclei occurred at 776 cm^{-1} and 834 cm^{-1}. The $C-O$ stretching of ester was observed between 1200 cm^{-1} and 1077 cm^{-1}. The carbonyl stretching occurred at 1689 cm^{-1}.

3.4. ^{1}H-NMR Spectra of Polymer

The ^{1}H-NMR spectrum of polymer **4** is shown in Fig. 4. The polymer shows signals between 6.8 ppm and 8.2 ppm due to aromatic protons. The assignable ethylenic proton of chalcone unit of polymer was obtained at 5.8 ppm and 6.2 ppm as doublet. Signal appeared at

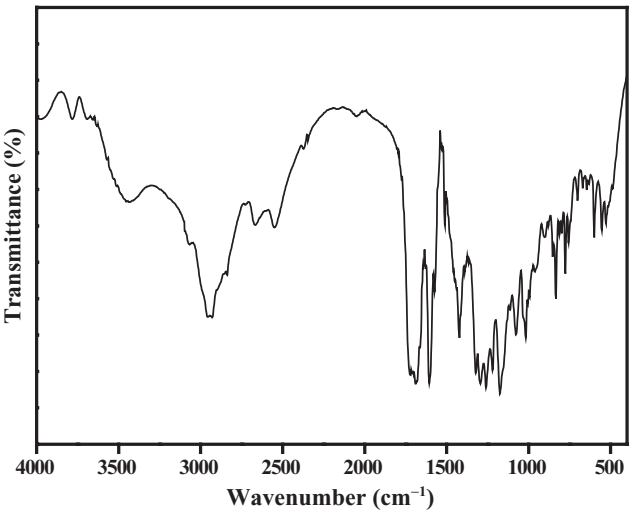


Figure 3. FT-IR spectrum of poly (MBSOEMA).

3.5 ppm due to methylenic protons. Signals at 3.8 ppm and 4.2 ppm were due protons in the $-CH_2-CH_2-$ group and terminal methoxy group. Signals at 1.03 ppm and 2.1 ppm were due to methyl and methylenic protons of polymeric backbone.

3.5. ^{13}C-NMR Spectra of Polymer

The proton-decoupled ^{13}C-NMR spectrum of poly (MBSOEMA) is shown in Fig. 5. The ketone carbonyl carbon resonance is observed at 189.5 ppm due to the presence of extended conjugations. The signals at 176.2 ppm are due to ester carbonyl carbon. The resonance of

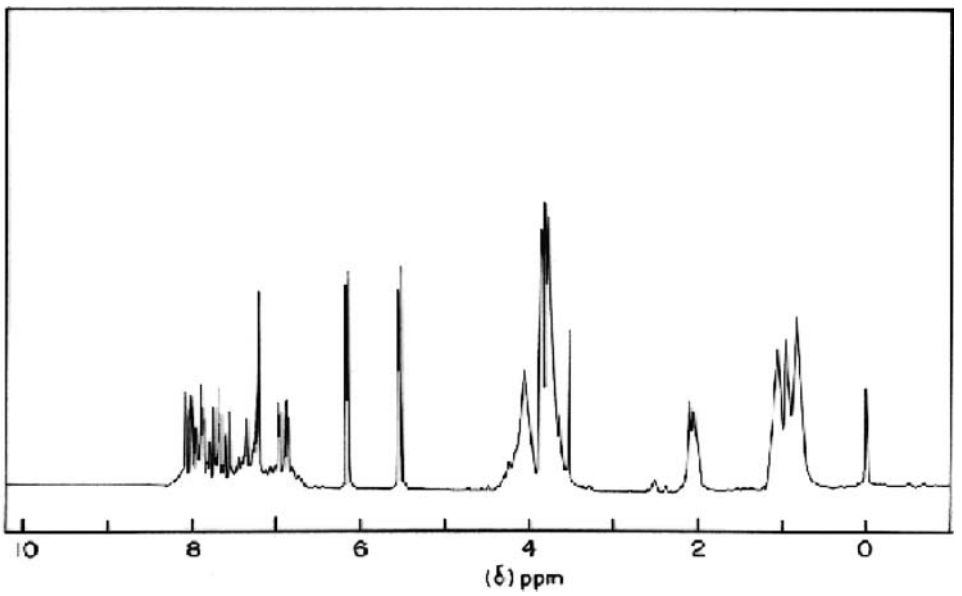


Figure 4. ^{1}H-NMR of poly (MBSOEMA).

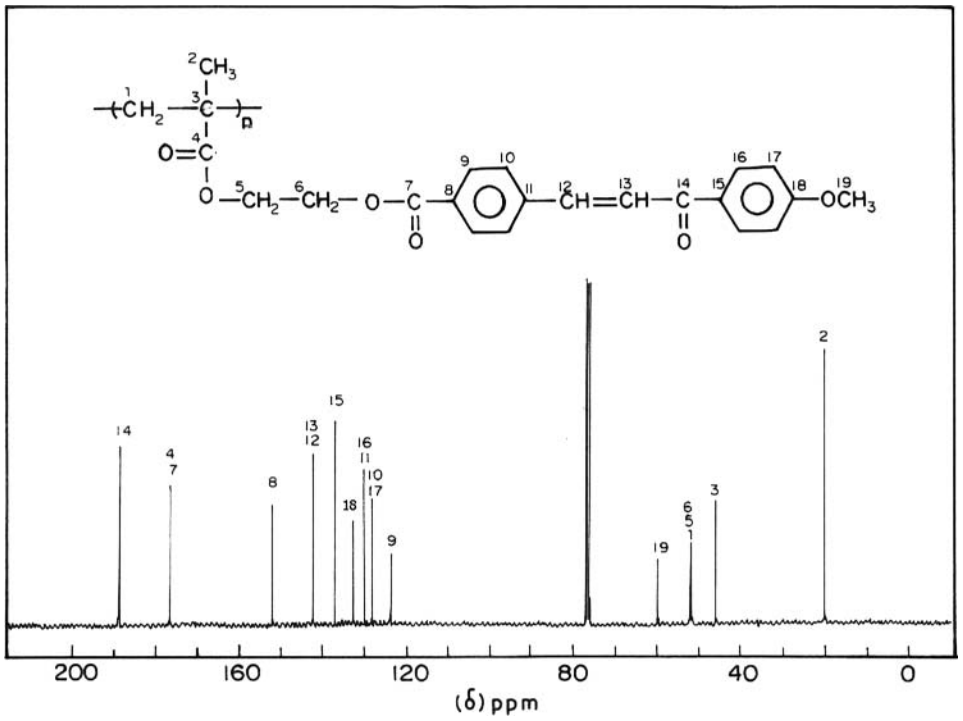


Figure 5. ^{13}C-NMR spectrum of poly (MBSOEMA).

aromatic carbons shows signals at 122, 160.5, and 118 ppm. The olefinic carbon signal was observed at 143.5 ppm. The backbone methylene and methyne carbon show signals at 52.5 and 45 ppm, respectively. The methoxy carbon resonates at 58.7 ppm. The α-methyl carbon appears at 18.9 ppm. The ethyl carbons in the polymeric unit show resonating signals at 50.5 ppm.

3.6. Thermal Study of the Polymer

Thermogravimetric analysis (TGA) of the polymer was carried out under nitrogen atmosphere in the temperature range of 20°C–600°C in order to investigate the thermal stability of the polymer. The TGA of the polymer is shown in Fig. 6. The homopolymer undergoes single stage thermal decomposition between 225°C and 475°C with the weight loss of 90%. The initial decomposition temperature (IDT) of polymer starts at 225°C. Due to the presence of ethylene spacer in the polymer backbone and pendant unit of chalcone showed weight loss of 50% and 75% at 390°C and 437°C, respectively. The thermal decomposition was almost completed at 450°C.

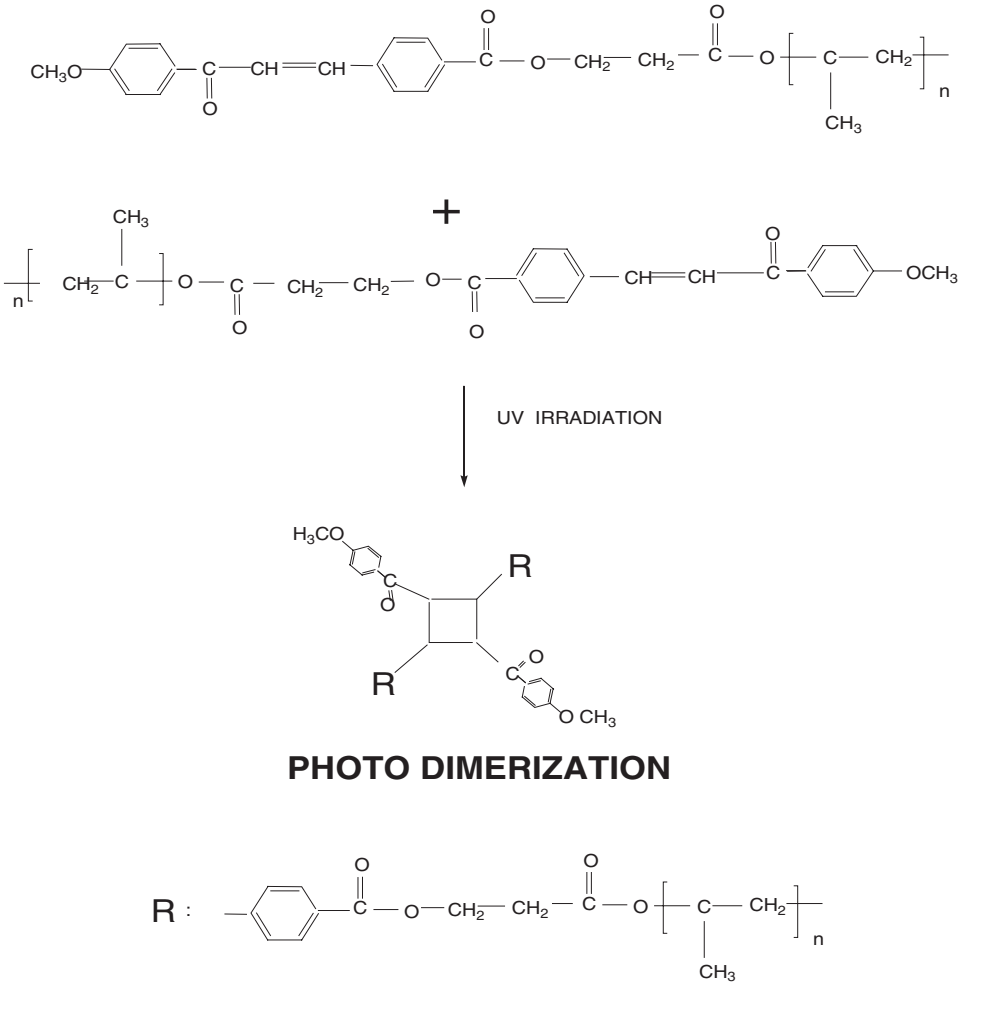
3.7. Molecular Weight

The number and weight average molecular weight of polymer **4** were determined by PL-GPC 650. The (M_n) and (M_w) values were 89,300 and 135,700 g/mole, respectively. The molar mass distribution of polymer given by polydispersity index (PDI) value was 1.52. The theoretical values of PDI for polymer produced by radical combination and

disproportionate were 1.5 and 2.0, respectively. The PDI value of photo-cross-linkable liquid crystalline polymer (PCLCP) was found to be 1.51 from the values of M_n and M_w, which suggests the strong tendency of chain termination by radical combination rather than disproportionation.

3.8. Photocrosslinking Properties

The photocrosslinking studies were carried out to study the changes occurred in the polymer during UV irradiation, which indicates photoresist nature of the polymer. The polymer solution was prepared in the concentration range of 10–20 mg/L using chloroform. It was irradiated with UV light of 254 nm and the photocrosslinking ability of the polymer was followed by the rate of disappearance of the C=C bond of photosensitive group in the UV spectrum. The photocrosslinking behavior of polymer **4** is shown in Scheme 3. The change in the absorption peak of polymer **4** is illustrated in Fig. 7. It is seen that the polymer



Scheme 3. Photocrosslinking of poly (MBSOEMA).

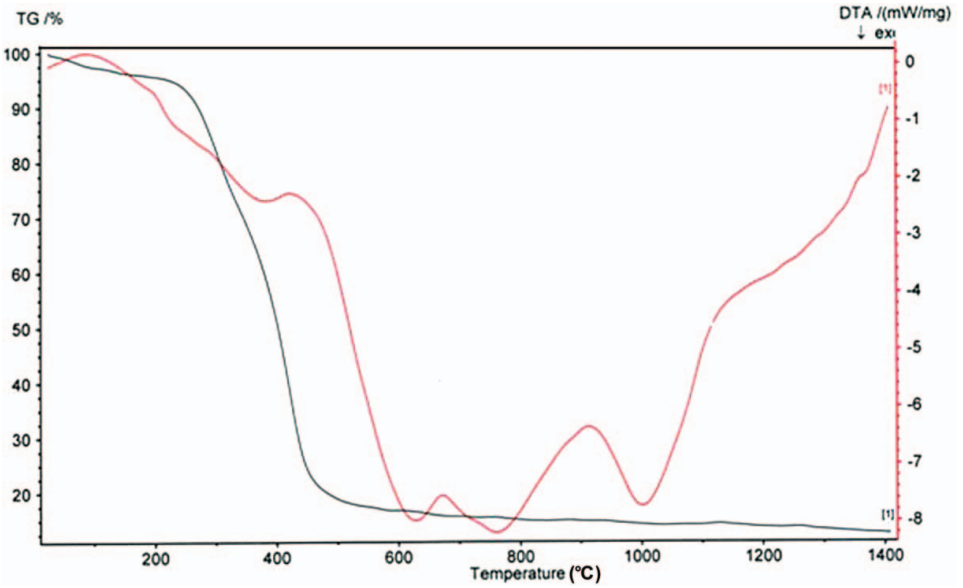


Figure 6. TGA analysis of poly (MBSOEMA) (Color figure available online).

containing chalcone moiety directly attached shows maximum absorption at 318 nm. The rapid photcrosslinking may be due to resonance effect produced by electron releasing methoxy group present in the pendant chalcone unit of polymer **4**. The photocrosslinking ability of polymer **4** was accelerated faster than π-π^* transition of pendant methoxy styryl

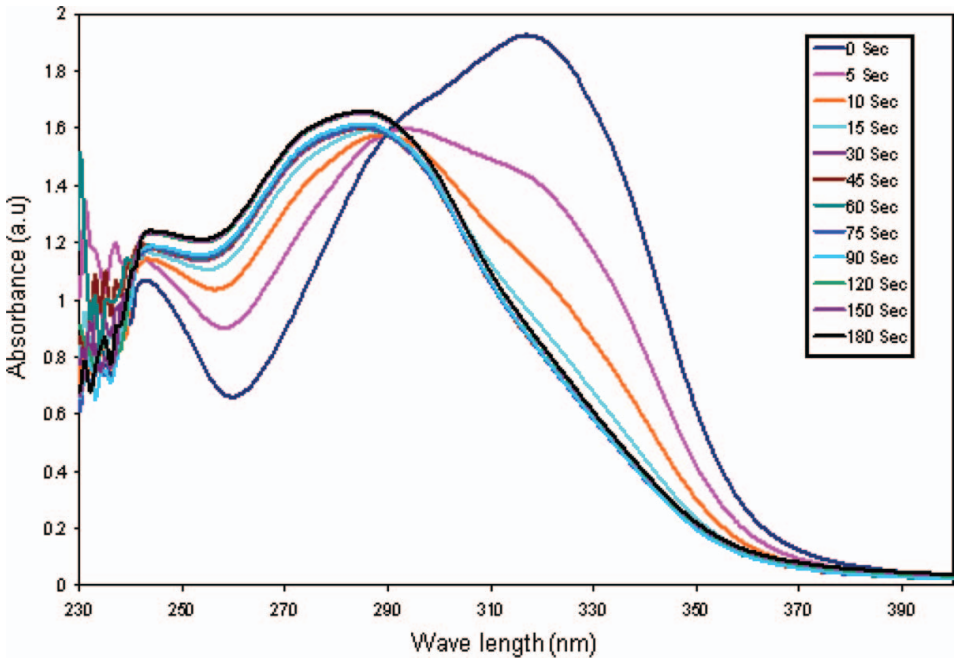


Figure 7. UV-Irradiation of poly (MBSOEMA) at various time intervals in chloroform solution.

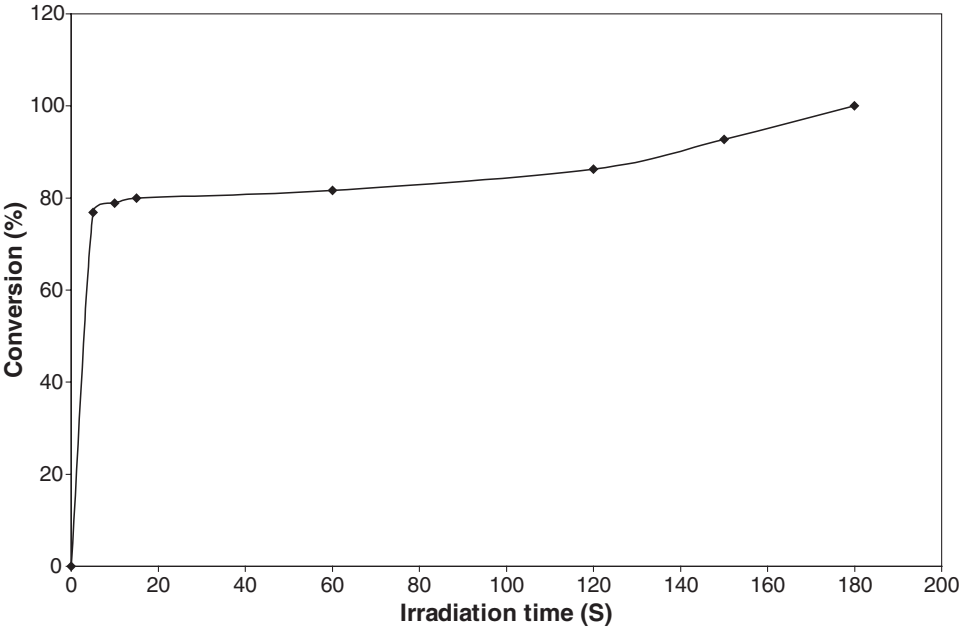


Figure 8. The disappearance rate of olefin double bond in the pendant chalcone unit with irradiation time.

group, which is evident from the appearance of isobestic point at 290 nm. The absorption intensity decreased rapidly with increasing irradiation time and band disappeared almost completely within 3 min of irradiation. The decrease in the UV absorption intensity is due to the crosslinking of polymer through $2\pi + 2\pi$ cyclodimerization of $-CH{=}CH-$ group of methoxy styryl group and leads to the formation of cyclobutane ring [23–25]. The photo-cross-linkable liquid crystalline homo polymer is insoluble in polar aprotic and chlorinated solvents, in which it was soluble before irradiation. The rate of disappearance of olefinic double bond in the pendant chalcone unit with irradiation time is shown in Fig. 8. Thus, the poly (MBSOEMA) with pendant chalcone moiety has a higher rate of photocrosslinking, even in the absence of sensitizers, leading to insolubility of the polymer. It is expected that this type of polymer might be useful as a negative photoresist for various applications.

3.9. Fluorescence Spectral Study

The photoresponsive nature of polymer **4** evidenced by fluorescence spectra is shown in Fig. 9. The fluorescence study of polymer was carried out by irradiation of the polymer in chloroform solution at 254 nm. The polymer was excited at a wavelength of 300 nm. Polymer **4** shows emission between 320 nm and 570 nm. It was observed from Fig. 9 that the fluorescence intensity showed drastic decrease after 10 s of irradiation and the decrease was continuous. This is because the electron releasing $-OCH_3$ group (donor) facilitates faster photocrosslinking and destroy the $\pi-$electron conjugation due to $2\pi + 2\pi$ cycloaddition reaction. The decrease in intensity continues until the formation of cyclobutane ring [26]. The fluorescence study reveals the indispensable use of polymer **4** in photoresist applications.

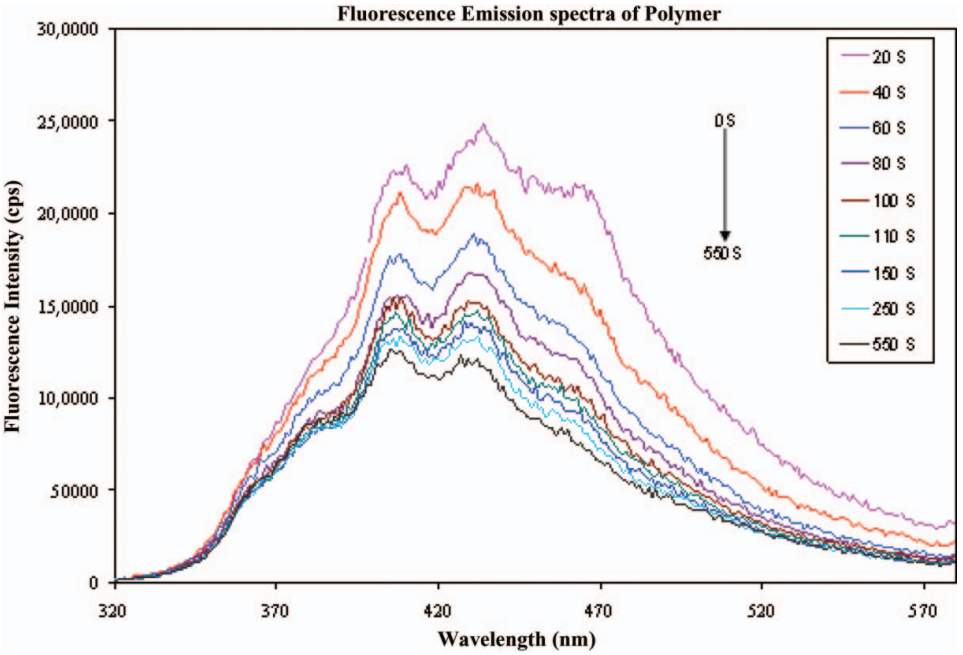


Figure 9. Fluorescence spectra of the poly (MBSOEMA).

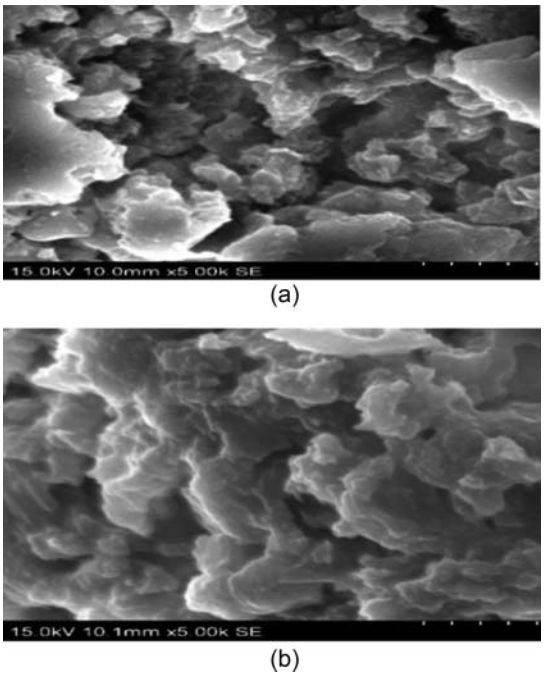


Figure 10. (a) SEM image of virgin polymer sample; (b) SEM image of photocrosslinked polymer.

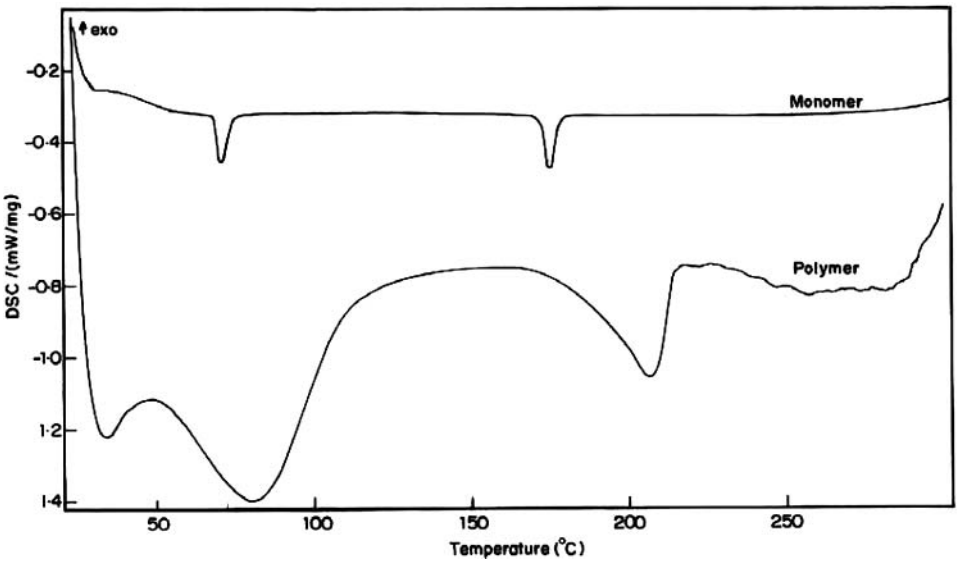


Figure 11. DSC thermogram of monomer and polymer.

3.10. Scanning Electron Microscopy (SEM)

The SEM technique can give high resolution images, which enables the visualization of morphological information without losing any accuracy during analysis. The synthesized photo-cross-linkable LCP poly (MBSOEMA) was irradiated with UV light of 254 nm for 30 min. The SEM analysis was carried out for both virgin polymer and photocrosslinked polymer samples. Figures 10(a) and (b) show morphology of virgin and photocrosslinked polymer samples, respectively. As observed from the SEM images in Figs. 10(a) and (b), the photocrosslinked polymer sample confirms loosely held dispsersion of polymeric materials, while virgin polymer exhibits compact stringent dispersion of polymeric surface.

3.11. Liquid Crystalline Properties

The development of photosensitive media based on liquid crystalline compounds for data recording, optical storage, and reproduction is one of the most rapidly developing areas in the physical chemistry of low molecular mass and polymer liquid crystals [27]. The rigidity of the mesogenic core, the flexible spacer length, and terminal units highly influence the molting temperature, mesophase temperature, and even molecular arrangement. The polymer shown in Scheme 2 is rod-like molecules and contain highly polar hindered

Table 1. Thermal and liquid crystalline properties of monomer and polymer

			DSC (°C)			HOPM (°C)		
Sample	Mesophase	T_g (°C)	T_m	T_i	ΔT	T_m	T_i	ΔT
Monomer	Grainy	50	75	174	99	73	171	98
Polymer	Grainy	53	80	208	122	84	210	126

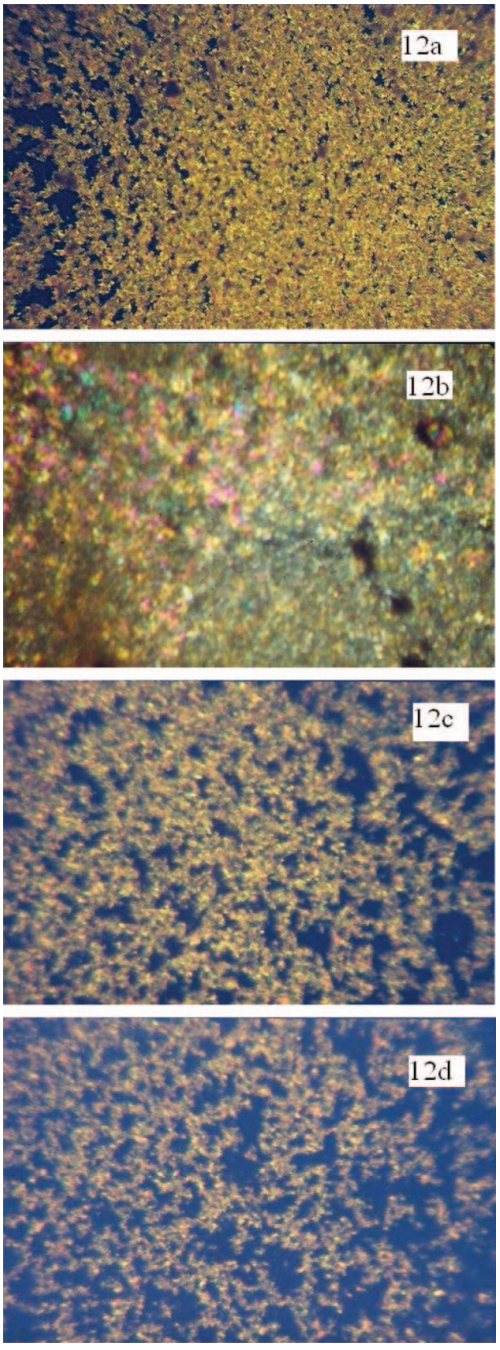


Figure 12. (a) Monomer shows liquid crystal phase from unidentified phase at 73°C; (b) monomer shows isotropic transition from grainy-like mesophase at 171°C; (c) poly (MBSOEMA) shows liquid crystal phase from unidentified phase at 80°C; (d) poly (MBSOEMA) at 208°C shows isotropic transition from mesophase.

pendant group revealed to have high interaction leading to the formation of liquid crystalline phases [28]. In LCPs, the polymer having rigid mesogen and shorter spacer should show higher transition temperature [29,30]; the presented novel LCP may show higher transition temperature due to the above-mentioned reason. The phase transition temperature and mesophase of this polymer were studied by the DSC thermogram and HOPM images. Generally, in the DSC thermogram, at the highest transition temperature there will be an endotherm corresponding to the transition from the LC phase to the isotropic phase. The transition in some cases from crystal to LC was marked by more than one endotherm. When such multiple curves were observed, the one having the highest temperature was attributed to the crystal to mesophase transition. The liquid crystalline properties of monomer and polymer were characterized by using DSC and HOPM studies. The DSC thermogram of monomer and polymer (Fig. 11) indicates that two endothermic transitions occurred on heating the monomer and polymer samples. The monomer sample showed lower transition at 75°C (T_m) whereas polymer sample showed at 80°C due to unidentified crystal to crystalline transformation. The higher endothermic transitions occurred at 174°C and 208°C for monomer and polymer, respectively. These higher endothermic transitions are due to transformation of grainy-like mesophase to isotropic transition. The thermal and liquid crystalline properties of monomer and polymer are shown in Table 1. The above recorded thermogram of monomer and polymer samples were compared with HOPM photographs shown in Figs. 12(a), (b), (c), and (d). The lower transition (T_m) that occurred at 73°C is shown in Fig. 12(a); it represents transition from the unidentified phase to the crystalline phase. Figure 12(b) shows grainy-like mesophase to isotropic at 171°C. In polymer **4**, it shows crystalline phase at 84°C (T_m) and grainy-like mesophase to isotropic transition at 210°C that are shown in Figs. 12(c) and (d). Both DSC and HOPM photographs showed negligible variations in their liquid crystalline properties.

4. Conclusions

The photo-cross-linkable LCP poly (MBSOEMA) was synthesized by free radical polymerization in THF using BPO as initiator. The synthesized polymer has been characterized by FT-IR, ^{1}H-NMR, and ^{13}C-NMR spectroscopy studies. The TGA analysis clearly indicates that the polymer shows good thermal and thermo-oxidative stability. The PDI value obtained from GPC indicates that the polymerization process was terminated by free radical recombination. The photocrosslinking study of the polymer was carried out at predetermined time interval by UV irradiation at 254 nm. From the UV study, the photoconversion of the polymer increases with increasing length of pendant chalcone unit in the polymer chain. The liquid crystalline property of the polymer was identified from the DSC thermogram as well as from HOPM studies. The UV studies reveal that the polymer may found application as a photoresist material. Since this polymer exhibits dual property of both liquid crystalline and photocrosslinking ability, it could be exploited in NLO applications [31].

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