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Main Chain Photoresponsive Liquid Crystalline Polymer synthesized through Hydrosilylation

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Abstract: Liquid crystalline siloxane azo polymers are synthesized through hydrosilylation reaction between diallyl azo monomer and siloxane dihydride. The molecular weights of the polymers ranged from 3000 – 4000 g mol⁻¹. The polymers were found to be soluble in chloroform, THF, DMF, DMSO, and DCM respectively. The photoresponsive behavior was studied during exposure of the polymers to UV radiation. The trans-cis photoisomerisation rate constants were also determined. The thermotropic behavior of the polymers was studied by using polarizing optical microscopy (POM) and differential scanning calorimetry (DSC). The polymers exhibited liquid crystalline texture of nematic droplets in POM images existing at room temperature and extending over broad temperature range up to 155 °C. The DSC thermogram also supported the characteristic liquid crystalline transitions.

Introduction

During the past decade thermotropic liquid crystalline (LC) polymers [1-3] have become an interesting research topic because of their applications in optical

data storage [4-6], piezo-, pyro-, and ferroelectric devices and elements requiring nonlinear optical characteristics [7] and so on. The high thermal stability and low glass transition temperature of polysiloxane has created interest in incorporating the polysiloxane backbone for liquid crystalline polymers (LCPs) [8-10]. Due to their low glass transition temperatures (T_g), the polysiloxane backbones are flexible, thereby the ordering of the attached LC's are not constrained. Such

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polymers having polysiloxane backbone also exhibit LC phase at ambient conditions [11-32]. Ueda and coworkers reported synthesis, characterization, and liquid crystalline behavior of thermotropic liquid crystalline polyimides with siloxane linkages having laterally substituted atoms and groups [33]. These pendant groups influenced the LC nature of the polymers. Smectic ordering in main-chain siloxane polymers and elastomers containing *p*-phenylene terephthalate mesogens was studied by Patil et al. [34]. Shim et al reported the synthesis and characterization of novel siloxane liquid crystalline polymers containing two symmetric mesogens [35]. Zhou et al worked on synthesis and properties of a series of mesogen-jacketed liquid crystalline polymers with polysiloxane backbones [36]. Interplay between nematic ordering and thermomechanical response in a side-chain liquid single crystal elastomer containing pendant azo mesogen units was also studied by Domenici et al. [37]. Mather et al described the synthesis of new siloxane-based main-chain LCs, including homopolymers, blends, and copolymers, with tunable clearing temperatures as governed by their chemical composition [38]. Liquid crystalline polyurethane thermoplastic elastomers with cyano-biphenyl mesogens pendant on the soft segment were synthesized for the first time by Hammond et al. [39]. The interplay between the liquid crystalline ordering of the elastomer and the morphology of the segmented copolymer was studied. Polymer liquid crystals with azobenzene moieties are promising materials for optical switching and image storage applications, because the orientation of mesogens in thin films of these materials may be modified by interaction of

azobenzene with light [40, 41]. Liquid crystalline polymers containing azobenzene groups are given special attention in recent years due to their potential application in photomechanical actuators, holographic recording medium, optical memory systems etc. [42-48]. The azobenzene chromophore has the ability to undergo changes in its conformation; from thermodynamically stable trans form to the less favored cis form, under UV radiation [49]. The trans-cis isomerization induces significant modifications both in geometry and in dipole moment of the chromophore enabling the azobenzene moiety to act as an optical switch for the photo control of polymeric structure where azobenzene is either connected in the side chain or incorporated directly in the main chain [50].

In our earlier publications both side chain and hyperbranched liquid crystalline polymers, with siloxane segment in the chain, have been reported and their photoactivity has been found to be encouraging [51-52]. To study the LC behavior and photoactivity of azo group placed in main chain containing siloxane segments, the synthesis was carried out through hydrosilylation between diene and dihydride monomers. The paper presents a study on the effect of siloxane chain length, LC behavior, and photoactivity of the synthesized polymers.

Experimental

Materials

p-Amino benzoic acid, phenol, sodium nitrite, allyl bromide, potassium carbonate were purchased from

S.D. Fine chemicals, India and used as received. Dimethylformamide (DMF), dichloromethane (DCM), petroleum ether (60–80 °C), ethyl acetate and toluene were purchased from Merck India Pvt Ltd. Toluene was distilled over sodium-ketyl radical. 1,1,3,3,5,5-hexamethyl trisiloxane, 1,1,3,3-tetramethyl disiloxane, platinum(0)-1,1,3,3-tetramethyl disiloxane complex (Karstedt's catalyst) purchased from Sigma-Aldrich were used without further purification.

Instruments and Techniques

¹H and ¹³C NMR spectra of the monomers and the polymers were recorded on a 500 MHz Bruker-FT NMR spectrometer using CDCl₃ as a solvent and tetramethylsilane (TMS) as an internal standard. The UV-Visible spectra of the polymers were taken by using Cary 500 Scan UV-Vis-NIR spectrophotometer. Leica DMLD optical polarizing microscope with image analyzer, equipped with LINKAM TMS 94 hot stage and LINKAM LNP controlling system, was used for studying the liquid crystalline behavior of the polymers. For photoresponsive studies the sample was irradiated with medium pressure 100W mercury lamp (Spectrolinker XL – 1500) in discontinuous mode from a distance of 10 cm for various time intervals.

Synthesis

The synthesis of 4-((4-hydroxyphenyl) diazenyl) benzoic acid (1) was carried out according to the procedure reported by Zhao et al. [46]

Synthesis of allyl 4-((4-(allyloxy)phenyl)diazenyl)benzoate

A solution of 4 - ((4-hydroxyphenyl) diazenyl) benzoic acid (1) (2 g, 8.26 mmol) in 25 mL DMF was taken in a R.B. flask. Potassium carbonate (5 g, 36.23 mmol) was added to the solution and heated at 80 °C for one hour. Allyl bromide (3 g, 24.8 mmol) was added drop wise to it over half an hour through rubber septum. The reaction was continued for 8 h and the reaction mixture was poured in to cold water. The aqueous solution was then extracted with dichloromethane. The organic layer was separated and washed with water and then passed through anhydrous sodium sulphate for removal of residual water and evaporated under vacuum to give the required compound. It was purified with column chromatography using 10% ethyl acetate in petroleum ether as eluent and later by solvent evaporation red colored solid was obtained.

Yield: 0.9 g, (35%); ¹H NMR (500 MHz, CDCl₃) δ: 4.72 (d, *J* = 5.0 Hz, 2H), 4.94 (d, *J* = 6.0 Hz, 2H), 5.39-5.56 (m, 4H), 6.11-6.19 (m, 2H), 7.12 (d, *J* = 9.0 Hz, 2H), 7.99 (d, *J* = 8.5 Hz, 2H), 8.03 (d, *J* = 8.5 Hz, 2H), 8.28 (d, *J* = 8.5 Hz, 2H); ¹³C NMR (125 MHz, CDCl₃) δ: 65.73, 69.04, 115.02, 118.12, 118.39, 122.33, 125.13, 130.61, 131.17, 132.11, 132.58, 147.05, 155.39, 161.64, 165.75.

Polymerization by hydrosilylation method

P1

Allyl 4-((4-(allyloxy) phenyl)diazenyl)benzoate (0.2 g, 1.82 mmol) was taken in a 25 mL two necked R.B. flask attached to schlenk line. Dry toluene (5 mL) was added to it through syringe and then karstedt's catalyst (0.012 ml) was added. Reaction mixture was heated to a temperature of 50 °C. 1, 1, 3, 3-tetramethyl disiloxane (0.16, 1.82 mmol) was added drop wise to it and the

reaction temperature was raised to 90 °C. Reaction was continued until the disappearance of Si-H peak in IR spectra at 2163 cm⁻¹. After completion of the reaction, the reaction mixture was cooled and precipitated in methanol to get viscous sticky mass. The material was dissolved in THF and reprecipitated in methanol thrice to get the final polymer, which was dried in a vacuum oven at room temperature under reduced pressure and used for further characterization.

Yield: (74%); ¹H NMR (500 MHz, CDCl₃) δ: 0.27 (br, 12H), 0.75 (br, 2H), 1.92 (br, 2H), 4.09 (br, 2H), 4.39 (br, 2H), 7.07-7.12 (br, 2H), 7.98-8.00 (br, 4H), 8.24- 8.32 (br, 2H); ¹³C NMR (125 MHz, CDCl₃) δ: 166.1, 162.3, 155.3, 146.8, 131.2, 130.5, 125.1, 122.3, 114.7, 70.8, 67.6, 23.1, 22.7, 14.1, 1.2, 1.1, 0.3.

P2

The synthesis of polymer P2 was done similarly as described for synthesis of polymer P1. In this case 1,1,3,3-tetramethyl trisiloxane was replaced with 1,1,3,3,5,5-hexamethyl trisiloxane (0.13 g, 0.62 mmol).

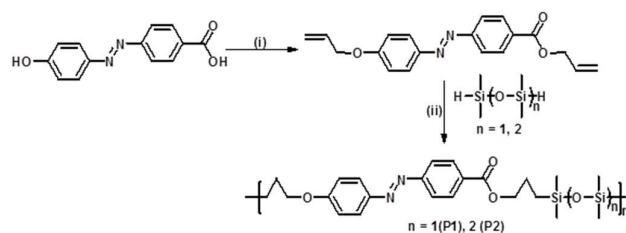
Yield: (74%); ¹H NMR (500 MHz, CDCl₃) δ: 0.15-0.21 (br, 18H), 0.75 (br, 2H), 1.92 (br, 2H), 4.09 (br, 2H), 4.39 (br, 2H), 7.07 (br, 2H), 7.98 (br, 4H), 8.24 (br, 2H); ¹³C NMR (125 MHz, CDCl₃) δ: 166.1, 162.3, 155.3, 146.8, 131.1, 130.5, 125.1, 122.2, 114.7, 70.8, 67.6, 23.1, 22.7, 14.1, 1.2, 1.0.

RESULTS AND DISCUSSION

Synthesis and Characterizations

Siloxane polymers containing LC moiety belong to a class of polymers in which orientation is quite easy due to flexible main chain. This tempted researchers to synthesize LC polymers in siloxane framework by adopting various strategies. Further, the glass transition being around or below room temperature, the LC polymers can have many interesting phase transition behavior.

Synthesis of silicone polymers, having azo group in the main chain, was carried out by hydrosilylation of azo-dialkene with hydride terminated siloxanes using karstedt's catalyst. The polymers are targeted for applications as photoactuators. The azo molecules are sufficiently rigid to behave as mesogens and the flexibility of siloxane provides higher degree of freedom so that mesogen rearrangement is facilitated. Further, siloxane backbone ensures fast photoresponse of the azo polymer. The synthesis of allyl 4-((4-(allyloxy) phenyl) diazenyl) benzoate was carried out through single step procedure leading to alkylation as well as esterification of 4-((4-hydroxyphenyl) diazenyl) benzoic acid (1) with allyl bromide in DMF as shown in **Scheme 1**. The overall yield of the final monomer is found to be 35%.



i) Allyl bromide, K₂CO₃, DMF, 80 °C; ii) karstedt's catalyst, toluene, 90 °C, 48h

Scheme 1: Schematics of Azo-dialkene synthesis and hydrosilylation polymerization

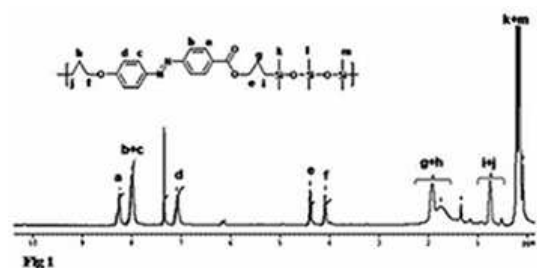


Figure-1: $^1\text{H-NMR}$ spectrum of polymer P2

The polymerization was carried out by taking equimolar ratio of the allyl 4-((4-(allyloxy)phenyl)diazanyl) benzoate and hydride terminated siloxanes in dry toluene in presence of karstedt's catalyst at 90°C . The reaction was monitored till the disappearance of Si-H peak around 2163 cm^{-1} confirming the completion of polymerization. The structure of polymers are confirmed from the broadening of peak in $^1\text{H-NMR}$ spectra (figure 1) as well as appearance of additional peaks due to reduction of allylic group of azo-dialkene. The $-\text{CH}_2$ peak of allyl group in azo dialkene at δ 5.39-5.56 ppm has shifted to δ 0.75 ppm. The $-\text{CH}$ peak of allyl group at δ 6.11-6.20 ppm has disappeared and formed $-\text{CH}_2$ appearing at δ 1.92 ppm. From GPC results, the molecular weights (M_n) of the polymers are found to be in the range of $3000\text{-}4000\text{ gmol}^{-1}$.

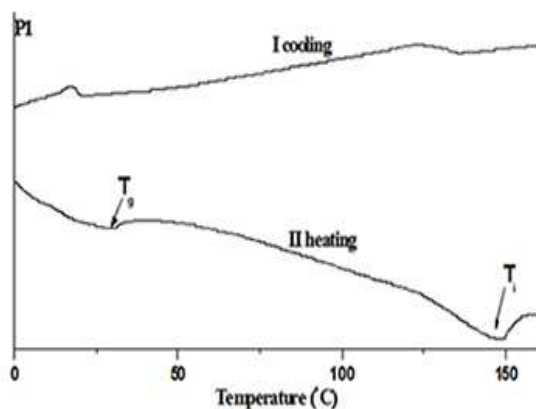


Figure-2: DSC Thermogram of Polymers P1

The idea of making low molecular weight is based on fast cis-trans interconversion and also fast mobility of segments so that photoactuator can be made using such type of polymer. We are working on siloxane backbone and these polymers are of low molecular weight. As a result, room temperature liquid crystallinity is observed.

Liquid crystalline property

DSC study

Figure 2 and 3 show the DSC thermograms of polymers P1 and P2 and the data are incorporated in Table 1. It is important to note that both the polymer exhibit endothermic peaks; the first one can be attributed to the amorphous to liquid crystalline (LC) phase transition and the second one is for LC to isotropic phase change. It is observed that amorphous to LC phase transition temperature for polymer P1 is higher as compared to polymer P2. This can be due to the fact that polymer P1 has smaller siloxane spacer compared to polymer P2, which is the reason for lower T_g of P2. The result is exactly reversed when the isotropization temperature of the polymers P1 and P2 are compared.

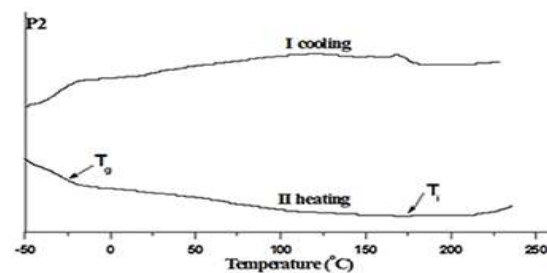


Figure-3: DSC Thermogram of Polymers P2

The isotropisation temperature of P2 is higher (155°C) as compared to polymer P1 (148°C). This indicates more ordering of mesogens in the polymer P2 as compared to polymer P1 due to presence of larger siloxane spacer. The cooling curve in DSC also demonstrates the isotropic to LC transition and LC to amorphous transition for both the polymers. The LC phase starts from 28°C and continues up to a temperature of 148°C in polymer P1 whereas it starts from -31°C and continues up to 155°C in polymer P2 indicating very broad LC phase for both the polymers.

POM Study

The mesogenic behaviors of the polymers were studied by POM. The POM micrographs are shown in Fig 4 and the observations are summarized in Table 1. Isotropization temperatures of P1 and P2 are recorded at 156 and 167°C respectively on heating the polymers at a rate of 10°C/min, in a hot stage. Then the samples were cooled at a rate of 2°C / min in which the appearance of droplet texture for P1 and schlieren texture for P2 at 140°C and 152°C respectively were observed, indicating presence of nematic phase (Fig 4a, b).

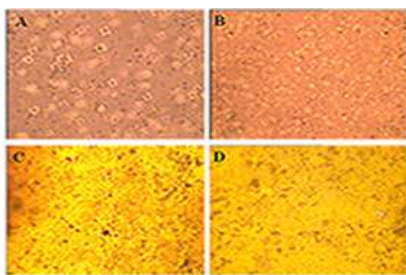


Figure-4: POM images showing nematic droplets texture for P1 (A) & (B) at 133°C and 30°C and schlieren texture of polymer P2 (C) & (D) at 30°C and 70°C respectively

Table 1: Physico-chemical properties of polymers

Polymer	Thermal transition (DSC) ^a	POM ^b T _i	M _n	M _w /M _n
P1	28 N 148	156	3000	1.23
P2	-31 N 155 I	167	3500	1.16

^a Transition temperature identified from heating cycle with DSC (at a heating rate of 10°C/min under N₂ atm); ^b transition temperature identified with POM at a heating rate of 5 °C/min; T_i= isotropization temperature.

This implies that the LC phase extends over broad temperature range for both the polymers. The POM transition temperatures observed are similar to that observed in DSC study.

Photo responsive Properties

Due to the presence of azo side groups in the polymers photoresponsive properties are generally expected. The chloroform solution of the polymer was kept in dark for three days to assume 100 % conversion to

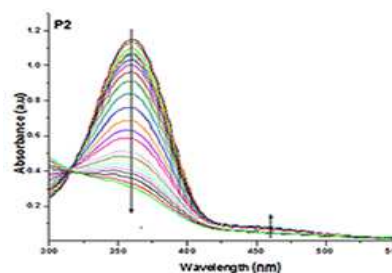


Figure-5: UV-Visible absorption spectra in chloroform solution for *trans-cis* isomerization of Polymer P2.

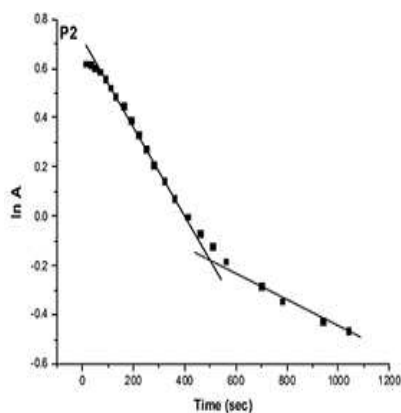


Figure-6: Plot of $\ln A$ vs. time for *trans-cis* isomerization of Polymer P2 in chloroform solution

trans form of azobenzene and then used for the photoresponsive study. The polymers show absorbance maxima (λ_{\max}) at 360 nm which is due to the π - π^* transition of stable *trans* form. Then the polymeric solutions were irradiated with UV light (365 nm) for different time intervals and the UV-Vis spectra were recorded. The decrease in intensity of π - π^* transition peak is observed which indicates *trans-cis* isomerization. Two absorbance peaks were observed in UV-Visible spectra for both P1 and P2. Representative spectra are shown in Fig. 5. The peaks around 360 nm and 450 nm represent π - π^* and n - π^* transitions respectively.

Table-2: The Rate constants of the polymers for *trans* – *cis* and *cis* – *trans* Isomerization

Polymer	λ_{\max} (nm) ^a π - π^*	λ_{\max} (nm) ^a n - π^*	K_x (sec^{-1}) ^b	K_y (sec^{-1}) ^c
P1	360	460	1.6×10^{-3}	5.51×10^{-4}
P2	360	460	1.8×10^{-3}	5.55×10^{-4}

^a recorded in chloroform solution; ^b rate constant calculated from the first slope of *trans* to *cis* isomerization graph ($\ln A$ vs. Time).

The absorbance vs. wavelength is plotted for different time interval of UV exposure. The rate constants of isomerization of the polymers are calculated from slope of the plot of $\ln A$ vs. time. Representative plot is shown in Fig 6. Table 2 shows the rate constants for *trans* to *cis* isomerization (K_x , K_y). Two slopes (K_x , K_y) are observed for all the polymers for *trans-cis* isomerization (Fig 6). It is found that the rate constant calculated from first slope of polymer P1 is lower than that of polymer P2. This may be due to higher flexibility of polymer P2 since it contains longer siloxane chain compared to P1.

Conclusion

Two types of siloxane polymers, possessing photoactive and liquid crystalline properties respectively, have been synthesized using hydrosilylation method. The polymers showed liquid crystalline characteristics existing over broad temperature range and also exhibited photoactivity. For polymer P2 schlieren texture and for polymer P1 a nematic LC droplets textures were observed. The rate constants for *trans* to *cis* isomerization increased with increase in the length of flexible siloxane spacer.

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