Liquid Crystallization of Poly(styrene-*co*-maleic anhydride) Induced by Intermolecular Hydrogen Bonds

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ABSTRACT: The liquid crystallization of general polymer (GP) with maleic anhydride in the main chain has been realized through molecular recognition and self-assembly based on intermolecular hydrogen bonds. Poly[styrene-*co*-(N-4-carboxylphenyl)maleimide] (SMIBA) was synthesized by imidization and dehydration of Poly(styrene-*co*-maleic anhydride) (SMA) with *p*-aminobenzoic acid (ABA) for use as an H-bonded donor polymer. 4-Methoxy-4′-stilbazole (MSZ) and 4-nitro-4′-stilbazole (SZNO$_2$) were prepared as an H-bonded acceptor. SMIBA was complexed with MSZ or SZNO$_2$ by slow evaporation from pyridine solution to form a self-assembly, which exhibits the mesophase, while neither of the individual components is mesogenic. The phase diagrams of a variety of mixtures between of SMIBA and stilbazoles have been established using DSC and POM. They show complete miscibility and high thermal stability of the liquid crystalline phase over the whole composition range. The tuning of liquid crystalline properties was achieved by changing the composition of the mixture and involving it with a mixture of SZNO$_2$ and MSZ. IR measurements strongly support the existence of an H-bonded complex between the carboxylic acid of SMIBA and the pyridine group of stibazoles. Unlike conventional side-chain liquid crystalline polymer (SLCP), supramolecular SLCP with a lower molecular weigh polymeric donor has higher thermal stability of the liquid crystalline phase due to the microphase separated in the hydrogen bonding case. Liquid crystallization of GP, such as SMA, induced by hydrogen bonds, offers a new route to prepare functional material with controlled molecular architecture from readily accessible and simpler precursors. © 1999 John Wiley & Sons, Inc. J Appl Polym Sci 74: 97–105, 1999

Key words: poly(styrene-*co*-maleic anhydride); liquid crystallization; hydrogen bonds; general polymer; supramolecule

INTRODUCTION

It has been recognized that liquid crystallization of general polymer (GP) is a new strategy to obtain functional and high-performance materials.[1–5] One is the ester exchange reaction between polyester and derivative of benzoic acid in melting[1–3]; the other is *in situ* composite between the general polymer and the thermotropic liquid crystalline polymer (TLCP).[4,5] But these approaches pose some restriction on the utility of the concepts on vinyl polymer in two counts. First, the ester exchange reaction was limited to polyester, such as PET, PBT, and PC. Second, the *in situ* composite requests a strong interaction between TLCP and GP to improve the compatibility. Unfortunately, these methods are not ideal routes for liquid crystallization of a vinyl polymer.

Recently, molecular architectures, through self-assembly, processes by specific molecular interactions, such as hydrogen-bond,[6–10] ionic,[11–13] ionic–dipolar,[14] and charge-transfer interaction[15] have been recognized to be a new strategy for building the molecular structure of side-chain liquid crystalline polymers, supramolecular SLCP. Hydrogen bonding is one of the most important means of interaction for these processes due to its stability and directionality. Although conventional side-chain liquid crystalline polymer consist of only covalent bonds, the supramolecular side-chain liquid crystalline polymer complex may contain an H-bonded mesogenic core or an H-bonded linker moiety connecting the mesogenic molecular to the polymer. Due to the simplicity of the preparation, supramolecular SLCP has an advantage in the tuning of the liquid crystalline properties[6] and design of the molecular structure, such as a supramolecular copolymer,[10] side-on SLCP,[8] network,[9] et al.

In general, supramolecular SLCP is also a mixture between polymer and low-mass weight molecule. It is not a simple mixture, but an orientation bled or programmed bled. Although built through a molecular recognition process, the system self-assembles to form supramolecular SLCP according to a program designed by nature or artificiality. Based on the concept of molecular recognition and self-assembly, we proposed a new approach to liquid crystallization of GP by the intermolecular hydrogen bond. This method involved two steps. First, the H-bonded donor was introduced into the side chain of the general polymer through conventional covalent bonds; second, the supramolecular host–guest complex between the polymeric moiety containing the H-bonded donor and nonmesogenic low-mass weight molecular containing the H-bonded acceptor was achieved by intermolecular hydrogen bonds.

SMA is a novel general copolymer.[16] Because of thermal properties and miscibility, SMA was used as a reagent in the blend of the polymer. Due to the reaction of maleic anhydride (MA), extensive attention has been made to the homopolymer and copolymer with MA in their main chain. The Schiff base-modified styrene–maleic anhydride copolymer was synthesized by the reaction of the SMA with the Schiff base, which was itself formed by a reaction of thiosemicarbazide with salicylaldehyde.[17] This modified SMA forms stable monolayer and good LB film with greater stability both mechanically and thermally. Ahlherim synthysized a new high molecular weight nonlinear optical polymer, prepared by the polymer analogous reaction of maleic anhydride with aminoalkyl functionalized azo dye.[18] Also, these kinds of polymer were used to form a supramolecular side-chain liquid crystalline polymer.[19] Poly(6-(4-octyloxyphenyl-azophenoxy)-hexyl maleicated) (PMAN-OAB) was prepared by esterization reaction between poly(maleic anhydride) and 6-(4-octyloxyphenyl-azophenoxy)-hexylalcohol. The complexes between PMAN-OAB and 4-(4-heptanoxybenzoyloxy)-4′-stilbazole (7SZ) exhibit mesogenic behavior with wide ranges of containing 7SZ in complexes.

In this article, SMA was modified through imidization with *p*-aminobenzoic acid and dehydration in order to introduction the benzoic acid moiety as a proton donor. Based on the concept of liquid crystallization induced by an intermolecular hydrogen bond, the supramolecular side-chain polymeric complexes between SMIBA as a proton donor and MSZ or SZNO$_2$ as a proton acceptor were prepared by slow evaporation from pyridine solution. Furthermore, the tuning of liquid crystalline properties was explored by changing the molar ratio of stilbazole in complexes and involving two or more stilbazoles. Two kinds of SMA with different molecular weights were modified through imidization and dehydration to study the effect of molecular weight of the polymer on mesogenic behaviors of complexes between modified SMA and stilbazoles.

EXPERIMENTAL

Materials

Unless stated otherwise, all the chemicals were of reagent grade. SMA is a random copolymer, synthesized by thermal copolymerization and obtained from the Institute of Shanghai Petrochemical (ISP); the composition of maleic anhydride is 13.7 or 17.0% (w/w) in copolymer, represented as SMA(A) and SMA(B), respectively.

Characterization

DSC measurements were performed on Perking-Elmer DSC-7; heating rates were 20°C/min in all case. First-order transitions (crystalline–mesogenic, crystalline–isotropic) were taken at the maximum point of the endothermic peaks; glass transition temperatures (T_g) were read at the midpoint of the change in the heat capacity. Po-

larizing optical macroscopic investigations were carried out with a JianNan XPT-7 optical microscope with a hot stage. IR measurements of all the samples were carried out on a Shimadzu IR-470 spectrophotometer. The element analysis of samples were carried out on Carlo Erba 1106. X-ray diffraction measurements were carried out on a Philips PW-1840 diffracometer with a 2KW Ni-filtered Cu Kα X-ray source. The samples were annealed by heating to their isotropization temperatures, followed by cooling to room temperature prior to X-ray diffraction studies.

Preparation of Poly(strene-*co*-(N-4-carboxylphenyl)maleimide) (SMIBA)

THF solution (250 mL) of 20.0 g SMA was heated to 76°C with stirring. Added slowly to this solution was 10.0 g *p*-aminobenzoic acid (ABA) in 80 mL THF. The solution was refluxed and stirred for another 24 h and was dropped into 2000 mL hot water with intensively stirring. The thin yellow solid was washed by hot water and ethanol three times. Thin yellow powder (21.3 g) was obtained after filtering and drying.

The 450-mL solution containing 20.0 g SMIA was heated to 140°C by an oil bath with stirring for 12 h. After cooling, the solution was dropped into a large amount of 0.1N hydrochloric acid. Thin yellow powder (18.3 g) was obtained after filtering and drying.

Preparation of Trans-4-Substituted-4′-Stilbazoles (XSZ)[20]

$$X = NO_2, OCH_3$$

Trans-4-substituted-4′-stilbazoles (XSZ) was synthesized by condensation reactions involving 4-picoline and 4-substituted-4′-benzaldehyde according to the modified method of Chiang.[20] Anisadehyde (24.2 mL) and 21.5 mL γ-methylpyridine were dissolved in 23.6 mL acetic anhydride. The solution was heated to 152–158°C, refluxed, and stirred for 12 h. After cooling, the solution was dropped into 1500 mL water, the brown precipitate was collected and washed with 1% NaOH 500 mL solution and hot water, respectively. The yellow crystal was obtained by filtering, drying, and recrystallization from ethanol. Yield 16%, T_m = 135°C. 4-Nitro-4′-stalbazole (SZNO$_2$) was prepared by the same method for MSZ. Yield 90%, T_m = 163°C.

Scheme 1

Preparation of Hydrogen-Bonded Complexes[6–10]

All hydrogen-bond complexes examined in the present study were prepared by the evaporation method from pyridine solution of the modified SMA and stilbazoles in appropriate proportions followed by slow drying in vacuum at 60°C. "Copolymer" complexes containing two different stilbazoles were prepared from SMIBA, and two different stilbazoles maintaining the exact 1 : 1 stoichiometry between the carboxylic acid unit of the SMIBA and the pyridine unit of the stilbazoles.

RESULT AND DISCUSSION

Modified SMA

From Scheme 1, the SMA, SMIA, and SMIBA contain anhydride, carboxyl, second and third amine, respectively. All those groups have characteristic absorb peaks in the IR spectroscope. Figure 1 shows the IR spectroscope of SMA(A), SMIA(A), and SMIBA(A). The sharp peak at 3000 cm^{-1}, which was due to the δCH, was changed into a wide peak from 2500–3500 cm^{-1}. This seems attributable to the dimer of carboxyl acid in SMIA. The characteristic peaks of maleic anhydride at 1780 and 1860 cm^{-1} (δCO) disappeared in SMIA, but the band at 1710 cm^{-1} appeared, which is the characteristic peak of carboxyl acid. The δNH at 1540 cm^{-1} shows at its place, which is the secondary amine. All these results show that maleic anhydride was changed into maleimide acid. After dehydration of SMIA, the IR spectroscope of SMIBA shows the characteristic peaks of maleimde at 1780 and 1710 cm^{-1} (δCO), 1540 cm^{-1} of secondary amine disappears, which

Figure 1 IR spectra of (a) SMA(A), (b) SMIA(A), and (c) SMIBA(A).

$$X = OCH_3, \quad NO_2$$

Figure 2 Structure of supramolecular side-chain liquid crystalline polymer built by hydrogen bonding between SMIBA and XSZ.

shows that the secondary amine (SMIA) was turned into the third amine (SMIBA).

The results of thermal analysis, element analysis, and intrinsic viscosity were summered in Table I. The glass transition temperature of the modified SMA through imidization, such as SMIA, is higher than that of SMA. This seems attributable to the introduction of a rigid moiety and interaction of carboxyl, which causes the increase of the rigidity of the main chain, so the T_g was increasing. Furthermore, SMIBA, the dehydration of SMIA, has a lower T_g, which is due to the SMIBA having only one carboxyl, but SMIA having two carboxyls.

Based on the Mark-Houwink equation, while polymer, solution, and temperature were fixed, the intrinsic viscosity was determined by the molecular weight of polymer. The intrinsic viscosity of SMIBA(A) is higher than that of SMIBA(B), so the molecular weight of SMIBA(A) is higher. From Table I, the intrinsic viscosity of SMIA and SMIBA is lower than that of SMA. This is due to curling of the main chain for the polymer in THF induced by the interaction of carboxyl.

For imidization and dehydration of SMA, according to 1 : 1 stoichiometry between anhydride of SMA and amino of ABA, the concentration of carboxyl in SMIBA can be calculated, which is 0.1176 mol/100 g or 0.1438 mol/100 g for SMIBA(A) or SMIBA(B), respectively. Compared with it, we can get the graft rate of the modified SMA.

Supramolecular "Homopolymer"

The supramolecular "homopolymer" complexes were obtained by molecular recognition and self-assembly process between the H-bonded donor SMIBA and the H-bonded acceptor MSZ. The structure of supramolecular SLCP built by hydrogen bonding is shown in Figure 2. SMIBA(A), the modified SMA through imidization and dehydration, was employed as an H-bonded donor. The stilbazole MSZ was complexed with the polymer.

Table I Modification of Poly(styrene-*co*-maleic anhydride) Through Imidization and Dehydration[a]

	$[\eta]^b$ (dL/g)	T_g (°C)	$N \%^c$ (w/w)	COOH (mol/100 g)	Graft %
SMA(A)	0.578	127.7			
SMIA(A)	0.521	155.0			
SMIBA(A)	0.474	151.5	1.3619	0.09728	82.76
SMA(B)	0.533	135.3			
SMIA(B)	0.462	167.1			
SMIBA(B)	0.417	162.0	1.5194	0.1085	75.48

[a] Imidization in THF at 76°C for 24 h, dehydration in DMF at 140°C for 12 h.
[b] Measurement in THF at 30°C.
[c] Elemental analysis of modified SMA.

Table II   Thermal Properties of H-Bonded SLCP Complexes Between SMIBA(A) and MSZ

MSZ in Complexes (mol %)			Phase Transition Behavior[a] (°C)			
10.99	G	111.1	LC	185.8	I
29.95	G	107.2	LC	197.1	I
50.02	G	93.6	LC	199.8	I
70.14	K	123.3	LC	171.2	I
89.99	K	126.0	LC	202.7	I

[a] Transition temperature (°C). G, glassy; K, crystal; LC, liquid crystalline; I, isotropic.

The thermal properties of the polymeric complexes are listed in Table II.

The glass transition temperature of the complexes is lower than T_g of SMIBA(A), and decreasing with increasing of the MSZ in the complexes. Furthermore, the complexes only exhibited melting transition without T_g when the molar ratio of MSZ is over 70%. From the supramolecular structures of SMIBA(A)/MSZ (Fig. 2), the hydrogen bond was formed between the carboxyl in SMIBA and the pyridine ring in MSZ. Due to the association, the carboxyls were "shielded" by the pyridine ring, the interaction of the carboxyl in the side chain of the polymer is weakened, and the T_g of the complexes decreases. The "shield" affection was increasing upon increasing of the MSZ in the complexes; this seems attributable to the decrease of T_g with the molar ratio of MSZ in the complexes. When the molar ratio of stilbazole is as high as 70%, besides the MSZ complexed with the polymer, there are some "free" MSZ, which aggregated each other to form the crystalline phase; so that complexes showed melting transition at heating, no glass transition was observed in this case. The melt point of the complexes is lower than that of MSZ.

The DSC and POM were employed to invest the liquid crystalline behavior. All complexes exhibit the stability mesogenic phase. From phase diagram of SMIBA(A)/MSZ (Fig. 3), the mesogenic stability (T_i) and mesomorphic range (ΔT) was increased with a molar ratio of MSZ in the complexes. For example, the complex 1 : 1 mol ratio of SMIBA(A)/MSZ displays a mesomorphase from 93.6 to 199.8°C. The supramolecular "homopolymer" may be considered as the copolymer polymerized by the mesogenic monomer and nonmesogenic monomer. While increasing of MSZ, the mesogenic monomer in the "copolymer" was increased. Due to the interaction of the mesogenic unit, the thermal stability of the mesogenic phase was increasing. For conventional SLCP, mesophase stabilization due to changes in composition of the copolymer is also observed. But synthesis of the copolymer with different composition by the convention method involves complicated preparative procedures, which is more difficult than by self-assembly.

The MSZ in the complex is over 50%, and the excess MSZ not only brought out the crystalline phase of complex, but also changed the texture of mesogenic. MSZ in the complexes is under 50%; the annealed samples exhibit schlieren texture at POM [Fig. 4(a)], but at over 70%, the annealed samples exhibit the fan texture [Fig. 4(b)]. So, the complex with the higher composition of MSZ shows a smetic phase and exhibits a nemetic phase in the lower composition of MSZ. That is the interaction of MSZ that did not complex with the polymer.

At the rage from 10–90%, the DSC curve of complex does not have the T_g of SMIBA and the melt transition peak of MSZ (Fig. 5). This is attributed to the association between the polymer and the low-mass weight molecule by an H-bond.

Figure 3 Phase diagram of the complexes formed between polymer SMIBA and MSZ.

(a)   (b)

Figure 4   Polarized photomicrographs of annealed H-bonded polymeric complex (a) SMIBA(A)/MSZ (50% : 50%), (b) SMIBA(A)/MSZ (30% : 70%).

The supramolecule prepared from two different components behaves as a single mesogenic component. Although, in some case the liquid crystalline transition were not observed during the cooling cycle, the mesomorphic transition was observed during the second heating of the samples.

IR measurements strongly support the existence of the H-bonded complexes. In Figure 6, the

Figure 5   DSC thermograms of (a) Polymer SMIBA, (b) MSZ and (c) their complex SMIBA/MSZ (90% : 10%).

Figure 6   IR spectra of (a) SMIBA(A), (b) MSZ, and (c) the H-bonded complex SMIBA/MSZ (1 : 1).

Figure 7 X-ray diffraction pattern of liquid crystalline SMIBA/MSZ (1 : 1 mol %) at annealed state.

Figure 8 Phase diagram of the complexes formed between polymer SMIBA and $SZNO_2$.

CO band at 1710 cm^{-1} observed for SMIBA, which was due to carboxylic acid dimers of the polymer SMIBA, is shifted to 1700 cm^{-1} in a 1 : 1 mol % mixture. This seems attributable to the complexes formed between the pyridine ring of MSZ and the carboxylic acid of SMIBA. The O—H bands observed at 2500 and 1900 cm^{-1} indicate that the weaker H-bond for the benzoic acid dimer was replaced by the strong H-bond between the carbolic acid of SMIBA and the pyridine ring of MSZ.

X-ray diffraction patterns of SMIBA/MSZ in the annealed state is shown in Figure 7, which exhibit the presence of only one crystalline peak corresponding to a d value of $4.5A^0$. It can be concluded that the complex appears to possess a nematic phase.

Different stilbazole has been selected as an H-bond acceptor to explore the effect of dipolar interaction. The transition temperature of the SMIBA/$SZNO_2$ are given in Table III. Figure 8

shows the phase diagrams of the supramolecular liquid crystalline polymer between SMIBA and $SZNO_2$. These complexes exhibit stable mesomorphic behavior. In particular, the complexes containing a strong polar nitro group display a multiphase transition, which was associated with the mesogenic phase transition. The mesogenic unit in the side chain in the supramolecule has a stronger polar interaction, which brings out the self-assembly to form a more order phase, such as the Smetic phase.

Compared to the T_i of SMIBA/$SZNO_2$ and SMIBA/MSZ, a slight decrease in isotropization temperature is observed for these polymeric complexes. This is different than Kato's work,[8] where we think there are two effects in action. One is the polar interaction of the nitro group in $SZNO_2$; it brings out the increase of the interaction between the mesogenic unit, which increases the stability of the mesogenicphase. The other is the absorb electron effect of nitro, which results in the decrease of the electron density in the pyridine ring, so the H-bond between the carboxylic acid and the pyridine is weaken; then the mesogenic stability

Table III   Thermal Properties of H-Bonded SLCP Complexes Between SMIBA(A) and $SZNO_2$

$SZNO_2$ in Complexes (mol %)	Phase Transition Behavior[a] (°C)						
11.16	G	99.4	LC_1	163.8	LC_2	193.2	I
29.97	G	99.8	LC_1	162.7	LC_2	189.6	I
49.96	G	100.9	LC_1	158.5	LC_2	188.4	I
70.10	K	146.2	LC_1	162.0	LC_2	186.1	I
90.06	K	151.8	LC_1	171.6	LC_2	205.9	I

[a] Transition temperature (°C). G, glassy; K, crystal; LC, liquid crystalline; I, isotropic.

Table IV Thermal Properties of H-Bonded SLCP Complexes Between SMIBA(A) and SZNO$_2$-MSZ

Mol Ratio of MSZ for H-Bond Acceptor (mol %)	Phase Transition Behavior[a]				
9.98	G	99.8	LC	189.6	I
28.72	G	102.7	LC	215.6	I
48.59	G	101.0	LC	219.7	I
69.47	G	102.0	LC	216.7	I
89.72	G	107.0	LC	180.6	I

[a] Transition temperature (°C). G, glassy; K, crystal; LC, liquid crystalline; I, isotropic.

decreases. Because SMIBA is a styrene and (N-4-carboxylphenyl)maleimide copolymer, the concentration of carboxyl in SMIBA is low, the concentration of SZNO$_2$ corresponding to the carboxyl of SMIBA in the complex is also low, and the absorb electron effect is dominant in this case.

Supramolecular "Copolymer"

To obtain a random "copolymer," the SMIBA can be simply complexed with a mixture of two or more different H-bond acceptors (see Fig. 9). In this study, stalbazoles containing electron donor and acceptor end groups have been used as components. Table IV gives the thermal properties of copolymeric complexes with the exact 1 : 1 stoichiometry between the carboxylic acid unit of SMIBA and the pyridine unit of the stilbazole. Figure 10 shows the phase diagrams of the liquid crystalline copolymer of SMIBA and a mixture of SZNO$_2$ and MSZ. In a range from 30–70% of MSZ in two H-bonded acceptors, the clearing temperature (T_i) and the mesomorphic range (ΔT) of the H-bonded complexes are higher than that of the corresponding homopolymeric complexes. For ex-

ample, the T_i of the complexes SMIBA(A)/SZNO$_2$: MSZ (50% : 25% : 25%) is 219.7°C, ΔT is 118°C; but the T_i of homopolymeric complexes SMIBA(A)/SZNO$_2$ and SMIBA(A)/MSZ are 188.4 and 199.8°C, respectively; ΔTs are 87.5 and 106.2°C, respectively. The increase of T_i and ΔT is attributed to the synergistic cooperative of the hydrogen bonding and electron donor–acceptor interaction, which leads to the mesospheric stability.

Effect of Molecular Weight

As an important component in the supramolecular SLCP, the polymer not only offers the H-bond donor, but also provides mechanical aid, which makes it possible to form a functional device with good mechanical properties. Unfortunately, most studies focused on the molecular design of the supramolecular SLCP; furthermore, the results in some cases appear to be somewhat confusing, due to different molecular weights of the polymer.[7]

To study the effect of molecular weights, two kinds of SMA were employed to modify through imidization and dehydration to act as an H-boned

Figure 9 Structure of the supramolecular side-chain liquid crystalline "copolymer."

Figure 10 Phase diagram of the complexes formed between polymer SMIBA(A)/(SZNO$_2$-MSZ).

Table V   Thermal Properties of H-Bonded SLCP Complexes for Different SMIBA with SZNO$_2$ (1 : 1 H-Bonded Complex)

	N %	Graft %	T_g	$[\eta]$	Phase Transition Behavior[a] (°C)					
SMIBA(A)	1.3619	82.76	153.3	0.474	G	100.9	LC$_1$	158.5	LC$_2$	188.4	I
SMIBA(B)	1.5194	75.48	159.6	0.417	G	109.3	LC$_1$	155.9	LC$_2$	211.1	I

[a] Transition temperature (°C). G, glassy; K, crystal; LC, liquid crystalline; I, isotropic.

donor polymer; the modified SMA were SMIBA(A) or SMIBA(B), respectively, in which SMIBA(B) has the lower molecular weight. The properties of the polymers and their complexes were summarized in Table V.

Compared with the complex between SMIBA and SZNO$_2$, the complex between SMIBA(A) and SZNO$_2$ has a lower T_i. This result is opposite to conventional SLCP, in which SLPC with a higher molecular weight has a higher T_i. In the supramolecular SLCP, the interaction between the polymer and low-mass weight molecule is hydrogen bonded; this association is not stronger than a covalent bond, so they may be microphase separated. The change of specific volume for the polymer with the molecular weight did not cause an increase of the density for the mesogenic unit in the mesophase. In contrast, the thermal motion of the main chain increases with the increase of the molecular weight of the polymer, which affects the order of the mesogenic unit in the complex. So the thermal stability of the supramolecular SLCP decreases and T_i is lower for the complex whose polymer component has a higher molecular weight in the hydrogen case.

CONCLUSION

We have developed a methodology to use molecular recognition and self-assembly via intermolecular hydrogen bonding for liquid crystallization of a general polymer. This new approach offers a relatively simple method to prepare novel functional materials with controlled molecular architecture from a general polymer, which is readily accessible and has simpler precursors.

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