

# Thermotropic Liquid-Crystalline Polymers: Synthesis, Characterization, and Properties of Poly(azomethine esters)

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**ABSTRACT:** A series of poly(azomethine ester) copolymers were synthesized by the solution polycondensation method with different diamines. The synthesized polymers were characterized by Fourier transform infrared spectroscopy, thermogravimetric analysis, differential scanning calorimetry (DSC), hot-stage polarized microscopy, wide-angle X-ray diffraction, and solution viscosity. All polymers showed good thermal stability. The thermotropic liquid-crystalline properties were examined by DSC and by microscopic observations. Except for one, all of the polymers

showed nematic liquid-crystalline behavior. The effects of temperature on crystallinity and the substituent on solubility, thermal stability, melting temperature, and viscosity were also studied. The voluminosity and shape factor were also computed from the viscosity data. © 2003 Wiley Periodicals, Inc. *J Appl Polym Sci* 88: 153–160, 2003

**Key words:** liquid-crystalline polymers (LCP); viscosity; differential scanning calorimetry (DSC)

## INTRODUCTION

Polymers incorporating mesogenic groups in the main chain are known to exhibit liquid-crystalline (LC) behavior. LC polymers consisting of a linear rigid mesogenic moiety (e.g., benzene ring, biphenyl group) and a flexible spacer in the main chain<sup>1–4</sup> are of interest due to their unique performance properties, namely, high mechanical strength and high-temperature fibers, laminates, and films.<sup>5,6</sup> In the past, many workers<sup>7,8</sup> have reported that the azomethine group with a flexible spacer is very useful in exhibiting LC properties. Chang and colleagues synthesized various thermotropic poly(amide azomethine ethers)<sup>9</sup> using flexible spacers and diamines, poly(amide azomethine) esters,<sup>10</sup> and poly(amide azomethine urethanes)<sup>11</sup> and studied their LC properties.

The purpose of this study was to synthesize various poly(azomethine esters) with rod-like segments connected by flexible spacers and to examine their liquid crystallinity and other properties. From earlier research work,<sup>12–15</sup> it is evident that the incorporation of azomethine groups in the main chain improve the thermal properties. We were interested in studying the effect of different diamine moieties on liquid crystallinity, thermal stability, and crystalline state for

these systems. Chang and colleagues<sup>10–16</sup> showed that the introduction of methoxy or ethoxy groups at the ortho position of the phenyl ring is helpful for lowering the transition and melting temperatures of such rigid rod-like polymers. Keeping these facts in mind, we introduced a methoxy group at the ortho position of the phenylene ring to examine the effects on melting temperature and liquid crystallinity. This article deals with the synthesis of different poly(azomethine esters) by the solution polycondensation method and characterization by different spectroscopic methods. Liquid crystallinity of these polymeric systems was also studied.

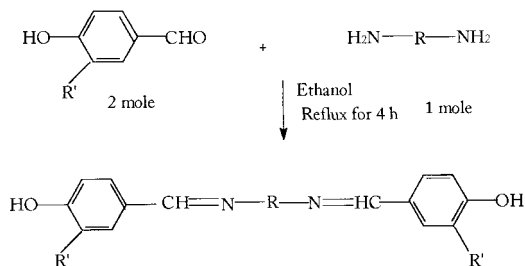
## EXPERIMENTAL

1,4-Phenylenediamine, 4,4'-diaminodiphenyl methane, benzidine, and adipoyl dichloride were purchased from Merck (Schuchardt, Germany). 4,4'-Diaminocyclohexyl methane (Lancaster, England) and *p*-hydroxybenzaldehyde (Sulab, Baroda, India) were used. *N*-methylpyrrolidone (NMP; Spectrochem, Bombay, India), ethyl alcohol (Baroda Chemical Industries Ltd., Baroda, India), and *n*-butanol (BDH Laboratory, Bombay, India) were used after distillation. Triethyl amine and 1,2-dichloroethane (Sulab) were distilled before use.

Elemental analysis of the monomers was performed with a Coleman C/H analyzer, and nitrogen was estimated by the Dumas method. Fourier transform infrared (FTIR) spectroscopy of the polymer samples was recorded by the KBr pellet method with a

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Scheme 1

Bomem-MB 104 (Canada) FTIR spectrophotometer. Thermogravimetric analysis (TGA) was performed with a Shimadzu DT-30 instrument at a heating rate of 10°C/min in air. Differential scanning calorimetry (DSC) measurements were conducted with a Mettler Toledo (Germany) instrument in aluminum pans at a heating rate of 10°C/min. Wide-angle X-ray diffraction (WAXD) of the polymer samples were recorded on Bruker D8 Advance instrument with Cu K $\alpha$  radiation. The optical textures of the polymer samples were studied with a Leitz laborlux 12 Pol (Germany) polarizing microscope fitted with a Kofler heating stage.

We synthesized the dihydroxyazomethine monomers by refluxing *p*-hydroxybenzaldehyde/vanillin with the appropriate diamine in the presence of ethanol for 4 h. The crude product was then filtered, dried, and recrystallized from *n*-butyl alcohol. The structures of the monomers were confirmed by elemental analysis and IR spectra. The general reaction equation and the structure of the various monomers are given in Scheme 1 (for R and R', see Table I).

The synthesis of all polymers was carried out by the solution polycondensation method. The synthesized

dihydroxyazomethine monomer (0.02 mol) was dissolved in 50 mL of NMP in a round-bottom three-necked flask fitted with a mechanical stirrer. Triethylamine (10 mL) was added, and the reaction mixture was stirred at room temperature in a water bath for 30 min. Adipoyl dichloride (0.02 mol) dissolved in 45 mL of dichloroethane was added dropwise to the reaction mixture at 0–5°C. After this addition was complete, the temperature was raised to 25°C, and the mixture was mechanically stirred for 5 h; thereafter, the mixture was poured into methanol (MeOH) to precipitate the polymer. The precipitated polymer was washed with MeOH, filtered, and dried at 80°C.

## RESULTS AND DISCUSSION

### IR spectra of monomers

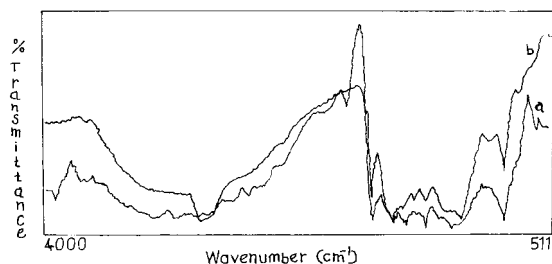
Bands at 1600 cm<sup>-1</sup> in AMM-2 and AMM-3 were characteristic of the —C=N— band. A band at 1380 cm<sup>-1</sup> in both spectra showed a bending vibration of the —OH. In both spectra, a band at 1680 cm<sup>-1</sup> was not observed, which indicated the absence of an aldehyde group and the formation of a —CH=N— linkage.

### FTIR

The representative FTIR spectra of PAM-2 and PAM-4 are shown in Figure 1. The characteristic bands of —C=N— group were found at 1598 and 1601 cm<sup>-1</sup>. Bands at 1749 and 1739 cm<sup>-1</sup> showed the carbonyl group (C=O) of esters, whereas bands at 739 and 839 cm<sup>-1</sup> showed C—H bending of the aromatic ring. In PAM-2, a band at 3234 cm<sup>-1</sup> showed weak vibrations

TABLE I  
Structure and Elemental Analysis of Monomers

Monomer	R	R'	Elemental analysis (%)					
			Calculated			Found		
			C	H	N	C	H	N
AMM-1		H	75.95	5.06	8.86	74.88	5.26	8.47
AMM-2		H	79.80	5.41	6.89	79.27	5.33	6.70
AMM-3		H	75.59	5.10	7.14	76.08	5.43	6.98
AMM-4		H	78.26	7.24	6.76	78.11	7.16	6.54
AMM-5		OCH <sub>3</sub>	72.23	5.16	6.57	72.02	5.38	6.28



**Figure 1** Representative FTIR spectra of (a) PAM-2 and (b) PAM-4.

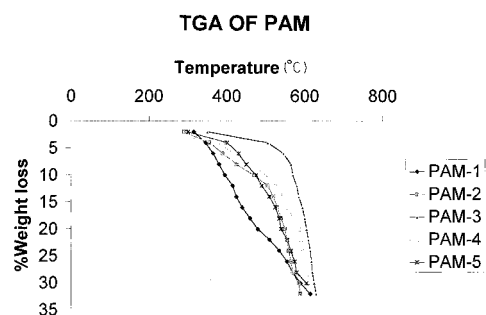
of the —OH group. Bands at 1377 and 1368  $\text{cm}^{-1}$  were characteristic of the bending vibrations of the —OH group.

### TGA

Thermal stability of the polymers was studied by TGA and DSC analysis. Thermogravimetry is a very useful method for measuring thermal stability and degradation kinetics.<sup>17</sup> TGA for all of the polymers is illustrated in Figure 2. All of the polymers showed single-stage decomposition and reasonably good thermal stability. The activation energy ( $E_a$ ) of decomposition was evaluated by the Broido method.<sup>18</sup> The equation used for the calculation of  $E_a$  was

$$\ln | \ln (1/Y) | = (E_a/R) (1/T) + \text{Constant} \quad (1)$$

where  $Y = (W_t - W_\alpha)/(W_0 - W_\alpha)$  and is the fraction of the number of initial molecules not yet decomposed and  $W_t$ ,  $W_\alpha$ , and  $W_0$  are the weights at any time  $t$  and at infinite time (residual weight at 600°C) and the initial weight, respectively, at a given temperature  $T$  (K). A plot of  $\ln[\ln(1/Y)]$  versus  $1/T$  gave a straight line (correlation coefficient = 0.95–0.99). The slope was related to  $E_a$ . The calculated values for the  $E_a$  of decomposition, the initial decomposition temperature (IDT), the maximum rate of decomposition ( $T_{\text{max}}$ ), and the complete decomposition (FDT) are listed in Table II. PAM-3 showed the highest  $E_a$  of decomposition,



**Figure 2** TGA plots of the polymers at heating rate of 10°C/min in air.

**TABLE II**  
Thermal Analysis Results of Poly(azomethine esters)

Polymer	IDT (°C)	$T_{\text{max}}$ (°C)	FDT (°C)	$E_a$ (kJ/mol)
PAM-1	315	415	590	41.3
PAM-2	290	555	600	51.8
PAM-3	350	618	630	188.7
PAM-4	235	605	615	40.9
PAM-5	300	575	605	73.7

and only 4% weight loss was observed at 500°C. This indicates that PAM-3 possessed the highest thermal stability among all of the polymers studied.

The thermotropic LC polyesters (e.g., Vectra, Xydar, polyesters prepared from terephthalic acid and substituted hydroquinone, polyesters prepared from bromoterephthalic acid and various naphthalene diols) are generally thermally stable. Their stabilities are not effected up to about 200°C. Over this temperature, depending on the particular polyesters, the stability may decrease. These poly(azomethine esters) were also thermally stable to the same extent as the previously mentioned polyesters.

### Thermotropic LC properties of polymers

The thermal behavior and thermotropic LC properties of the polymers were studied by DSC and hot-stage optical polarized microscopy. DSC and optical microscopy were used to examine the phase behavior of the polymers, and the data are summarized in Table III. In DSC, PAM-1, PAM-3, PAM-4, and PAM-5 showed a mesomorphic transition ( $T_m$ ) at 308, 321, 174, and 175°C, respectively. PAM-1 showed one broad endotherm at 305°C and a second one at 410°C, whereas PAM-5 showed two endotherms at 175 and 220°C. The first one could be attributed as  $T_m$  and the second one as the isotropic transition ( $T_i$ ). Representative DSC curves of PAM-4 and PAM-5 are shown in Figures 3(a) and 3(b), respectively. Thus PAM-5 melted at a lower temperature than PAM-1, indicating that the substitu-

**TABLE III**  
Transition Behavior of Poly(azomethine esters)

Polymer	LC phase <sup>a</sup> (°C)	Transition temperature <sup>b</sup> (°C)	
		$T_m$	$T_i$
PAM-1	K 306 N	308	410
PAM-2	K 270 N 280 I	—	—
PAM-3	—	321	—
PAM-4	K 170 N 212 I	174	—
PAM-5	K 172 N 240 I	175	220

K, crystalline; N, nematic; I, isotropic.

<sup>a</sup> As under the polarizing microscope.

<sup>b</sup> From DSC, first heating, 10°C/min.

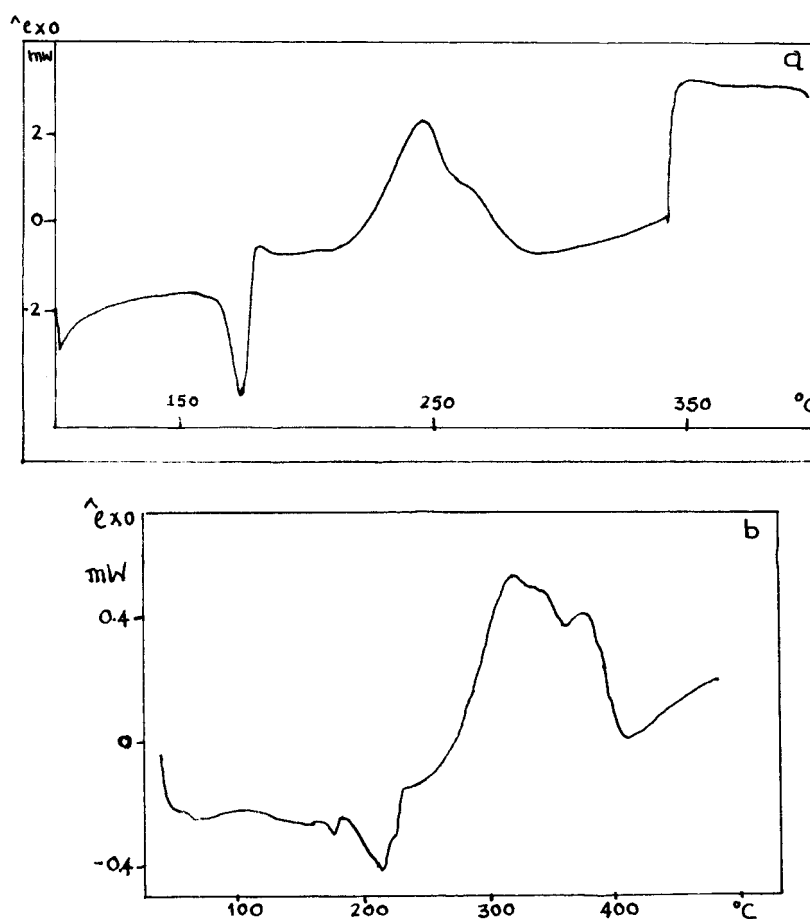


Figure 3 Representative DSC thermograms of (a) PAM-4 and (b) PAM-5.

tion of a  $-\text{OCH}_3$  (methoxy) group was useful in bringing down the melting temperature of the polymer. This was due to the fact that substituents could act to reduce the coplanarity of adjacent mesogenic groups and increase the diameter or decrease the axial ratio of the mesogens.<sup>10</sup> Several workers, namely, Li and Chang,<sup>16</sup> Meurrisse et al.,<sup>19</sup> and Sek,<sup>20</sup> have used different groups (substituents) in past to bring down the  $T_m$  and  $T_i$  of LC polymers.

PAM-4 showed the lowest  $T_m$  and  $T_i$  in optical polarized microscopy, which may have been due to the cyclohexyl moiety in the polymer backbone. Thus, the introduction of substituents or a planar cyclohexyl moiety could be used in the rigid polymer backbone in place of a benzene or benzidine moiety to bring down the  $T_m$  and  $T_i$  of polymer systems.

Thermotropic LC behavior was also studied by hot-stage polarized microscopy. Except for PAM-3, all polymers showed nematic LC behavior in polarized microscopy. In the DSC run, PAM-3 showed an endotherm at 321°C, which might have been due to a crystalline to  $T_m$  transition, but in hot-stage microscopy, we did not observe such a mesophase transition up to 300°C. PAM-1 and PAM-2 showed a banded

spherulitic fan-shaped texture (Fig. 4), whereas PAM-4 showed a droplet-type nematic texture (Fig. 5). PAM-5 showed a Schlierian texture of nematic LC phase above 170°C.

#### WAXD

WAXD patterns of poly(azomethine esters) are shown in Figure 6. All polymers showed semicrystalline be-

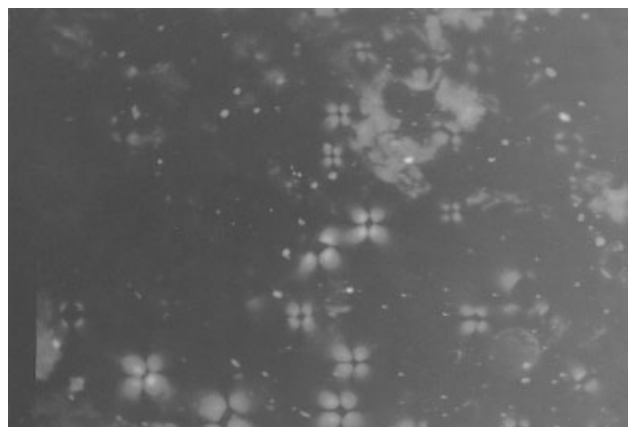


Figure 4 Polarizing microphotograph of PAM-2 at 275°C.

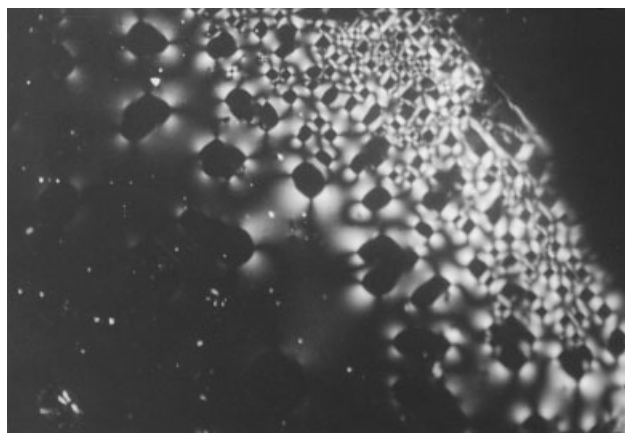


Figure 5 Polarizing microphotograph of PAM-5 at 175°C.

havior. Except for PAM-3, all polymers showed a peak at  $2\theta \cong 20^\circ$ . PAM-3 showed less crystallinity compared to others and showed weaker diffraction peaks around  $2\theta = 20^\circ$ .  $d$ -spacing and Bragg angles of the polymers are given in Table IV. PAM-1 showed two main diffraction peaks at  $2\theta = 20.18$  and  $21.50^\circ$ , whereas PAM-2 showed one very intense peak at  $2\theta = 20.33^\circ$ . PAM-3 did not show any intense peak at all, and only a weak diffused halo was found at  $2\theta = 8^\circ$  and  $2\theta \cong 20^\circ$ . PAM-4 showed two small-angle peaks at  $2\theta = 2.25$  and  $4.50^\circ$  apart from a few peaks at  $2\theta = 17, 18,$  and  $22^\circ$ , whereas PAM-5 showed two sharp peaks at  $2\theta = 18$  and  $20^\circ$  and two medium peaks at  $2\theta = 24$  and  $25^\circ$ .

The crystallinity of the samples were calculated with the following equation<sup>21,22</sup> (Table IV):

$$\% \text{ Crystallinity} = I_c/I_a + I_c \times 100 \quad (2)$$

where  $I_c$  is intensity of the crystalline component and  $I_a$  is the intensity of the amorphous component.

The effect of temperature on the crystallinity of the polymer samples was also studied. Figures 7 and 8 show WAXD patterns of PAM-4 and PAM-1 at different temperatures. At room temperature (25°C), PAM-4

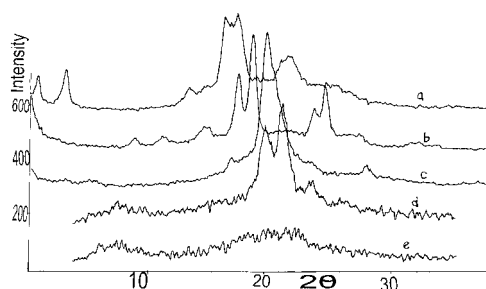


Figure 6 WAXD diffractograms of polymer samples at room temperature: (a) PAM-4, (b) PAM-5, (c) PAM-2, (d) PAM-1, and (e) PAM-3.

TABLE IV  
Observed X-Ray Diffraction Data of the Polymers

Polymer code	$2\theta$	$d$ -spacing (Å)	Intensity (%)	Degree of crystallinity (%)
PAM-1	20.18	4.40	85	20
	21.50	4.13	100	
	23.88	3.72	42	
PAM-2	17.54	5.05	23	36
	20.33	4.36	100	
	28.07	3.17	19	
PAM-3	8.55	10.33	71	11
	20.34	4.36	99	
	21.78	4.07	100	
PAM-4	2.25	39.13	41	24
	4.50	19.60	47	
	14.24	6.21	30	
	17.09	5.18	97	
	18.03	4.91	100	
PAM-5	22.01	4.03	60	25
	9.95	8.88	15.8	
	12.17	7.26	15.7	
	15.5	5.71	24.4	
	18.08	4.9	68.2	
	19.20	4.62	100.0	
	23.98	3.71	38.6	
	24.84	3.58	58.1	

showed sharp peaks at around  $2\theta = 18^\circ$  and a medium broad peak at around  $2\theta = 21^\circ$ . With an increase in temperature, these peaks became broader, and at  $225^\circ\text{C}$ , both peaks disappeared, and only halo was seen. This indicates that at isotropic temperature ( $T_i = 220^\circ\text{C}$ ), PAM-4 behaved in an amorphous manner. Similar patterns were observed by many workers<sup>23,24</sup> at higher temperatures for thermotropic LC polymers. Stocker et al.<sup>25</sup> reported for LC polymers that at isotropic temperatures, they showed diffuse broad peaks at  $2\theta \cong 20^\circ$  and a small-angle peak at  $2\theta < 5^\circ$  vanished. Similar behavior was observed in the case of PAM-1. It showed two main diffraction peaks at  $2\theta = 20.18$  and

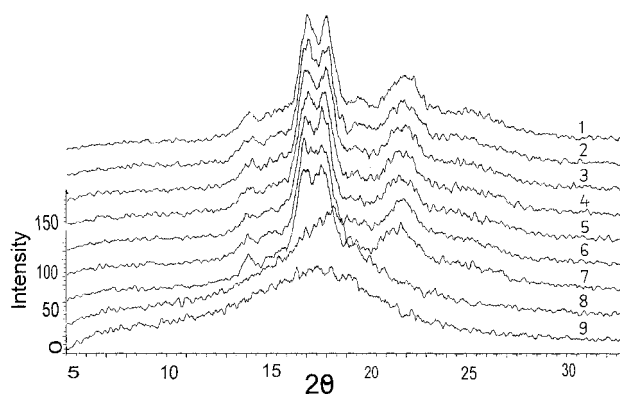
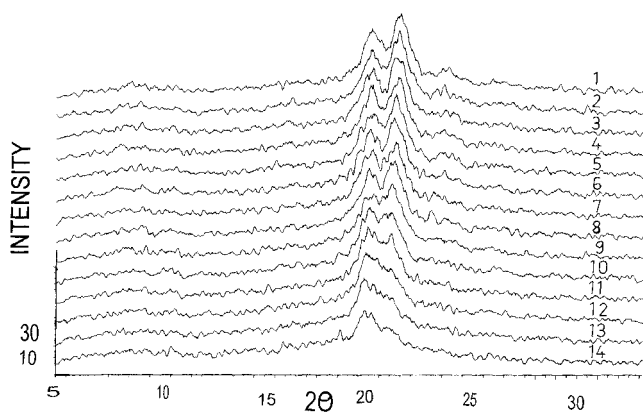


Figure 7 WAXD of PAM-4 at different temperatures: (1) 25, (2) 50, (3) 75, (4) 100, (5) 125, (6) 150, (7) 175, (8) 200, and (9)  $225^\circ\text{C}$ .



**Figure 8** WAXD of PAM-1 at different temperatures: (1) 25, (2) 50, (3) 75, (4) 100, (5) 125, (6) 150, (7) 175, (8) 200, (9) 225, (10) 250, (11) 275, (12) 300, (13) 325, and (14) 350°C.

21.50°. As the temperature increased, both peaks became broad, and at 300°C, the peak at 21.50° vanished.

The crystallinity of the synthesized polymers were compared with some common LC polyesters (e.g., copolyesters containing terephthaloyl chloride, a cycloaliphatic spacer, and 2-chlorohydroquinone; polyesters containing 4-acetoxybenzoic acid, hydroquinone diacetate, and terephthalic acid (TPA); polyesters prepared from TPA, a *m*-hydroxybenzoic acid unit, etc.). Literature values show that these polyesters were semicrystalline. Their *d*-spacing values were in the range of 3.0–5.0 Å, and they showed medium/sharp peaks around  $2\theta = 15^\circ$  and between  $2\theta = 20$  and  $25^\circ$ . The synthesized poly(azomethine esters) had similar crystalline behaviors, as can be seen in Table IV.

### Solubility

The solubility characteristics of the poly(azomethine esters) were tested in various solvents, including  $\text{CHCl}_3$  (chloroform), MeOH, NMP, dimethylformamide (DMF), dimethylsulfoxide (DMSO), *p*-chlorophenol (PCP), and concentrated  $\text{H}_2\text{SO}_4$ . A 5% (w/v) solution was taken as a criterion for solubility. All polymers were insoluble in acetone, chloroform, and MeOH. PAM-4 showed very good solubility in NMP, DMF, PCP, and concentrated  $\text{H}_2\text{SO}_4$  due to the cyclo-

hexane structure in its polymer backbone. PAM-1 showed solubility in PCP and concentrated  $\text{H}_2\text{SO}_4$  and was not completely soluble in NMP, DMF, and DMSO, whereas PAM-5 showed better solubility in DMF and NMP compared to PAM-1 (Table V). This indicates that a  $-\text{OCH}_3$  (methoxy) group was helpful in increasing solubility, which means that the substitution of groups on the polymer backbone was very useful in enhancing the solubility of the polymer samples.<sup>26–28</sup>

Some reported LC polyesters [e.g., polyesters prepared from hydroquinone and various aryloxyterephthalic acids, copolyesters containing spirobicomane moieties, copolyesters containing 2-( $\alpha$ -phenyl isopropyl)hydroquinone moiety] were soluble in PCP, 1,1,2,2-tetrachloroethane, NMP, DMF, trifluoroacetic acid (TFA)/chloroform (1:1 v:v), and so on. The synthesized poly(azomethine esters) were also soluble in NMP, DMF, PCP, and other organic solvents.

### Viscosity

The viscosity behavior of the polymers was studied at different temperatures (45, 50, and 55°C) with PCP as a solvent. The intrinsic viscosity ( $[\eta]$ ) was calculated with the Huggins and Kreamer equations<sup>29,30</sup> (Table VI). By plotting  $\eta_{\text{sp}}/\text{concentration}$  ( $C$ ) versus  $C$  and  $\ln\eta_r/C$ , we computed  $[\eta]$  (Fig. 9), where  $\eta_{\text{sp}}$  and  $\eta_r$  are specific and relative viscosity, respectively.  $[\eta]$  was found to decrease with increasing temperature for all the polymer systems. There was no significant change in the intrinsic viscosities of PAM-1 and PAM-5, which indicates that substitution of the  $-\text{OCH}_3$  group on the benzene ring in the polymer backbone had no effect on the solution viscosity. However, these polymers had much lower  $[\eta]$  ( $[\eta] = 0.20\text{--}0.28$  dL/g in PCP) than some LC polyesters [e.g., polyesters prepared from 2-( $\alpha$ -phenylisopropyl)hydroquinone moiety (inherent viscosity = 0.78–2.79 in PCP)], which was the only important difference in the properties studied of the synthesized poly(azomethine esters) from that of polyesters.

Relative viscosity data at different  $C$ s were used for the calculation of the voluminosity ( $V_E$ ) of the polymer

**TABLE V**  
Solubility Study of Polymers

Polymer	$\text{CHCl}_3$	MeOH	NMP	DMF	DMSO	PCP	Concentrated $\text{H}_2\text{SO}_4$
PAM-1	—	—	±	±	±	+	+
PAM-2	—	—	+	+	+	+	+
PAM-3	—	—	—	—	—	+	+
PAM-4	—	—	+	+	±	+	+
PAM-5	—	—	+	+	+	+	+

— = Insoluble; + = soluble; ± = partially soluble; +\* = soluble on heating.

TABLE VI  
Viscosity Data of the Polymers at Different  
Temperatures in Different Solvent Systems

Polymer	Temperature (°C)	$[\eta]$ (dL/g)	$V_E$ (dL/g)
PAM-1	45	0.281	0.11
	50	0.258	0.103
	55	0.232	0.092
PAM-2	45	0.253	0.100
	50	0.237	0.094
	55	0.219	0.087
PAM-3	45	0.245	0.097
	50	0.221	0.088
	55	0.205	0.081
PAM-4	45	0.263	0.104
	50	0.243	0.096
	55	0.220	0.087
PAM-5	45	0.282	0.113
	50	0.257	0.102
	55	0.234	0.094

solutions at a given temperature. We obtained  $V_E$  by plotting a quantity  $\chi$  against  $C$  (g/dL), where

$$\chi = (\eta_r^{0.5} - 1)/C (1.35 \eta_r^{0.5} - 0.1) \quad (3)$$

The straight line obtained was extrapolated to  $C=0$ , and the intercept yielded  $V_E$  (Table VI).  $V_E$  (dL/g) is a function of temperature and is a measure of solvated polymer molecules.  $V_E$  showed a decrease with increasing temperature for all of the polymer systems, which matches our earlier work.<sup>31</sup> Narang and Garg<sup>32</sup> calculated  $V_E$  for bovine serum albumin, hemoglobin, and egg albumin with eq. (3), which enabled them to determine the size and shapes of near-spherical macromolecules in solutions.

The shape factor ( $\nu$ ) was also calculated for all polymer systems from the following equation:

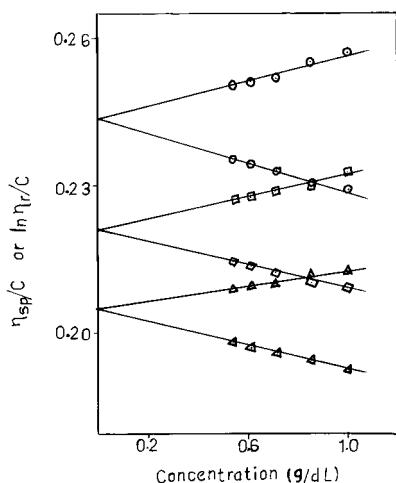


Figure 9 Plot of (1)  $\eta_{sp}/C$  and (2)  $\ln \eta_r/C$  against  $C$  of PAM-3 at different temperatures: (○) 45, (□) 50, and (△) 55°C.

$$[\eta] = \nu \cdot V_E \quad (4)$$

$\nu$  gives an idea about the shape of the macromolecules in the solution.<sup>33</sup> It was found to be  $2.5 \pm 0.02$  and was independent of temperature. This indicates that the polymers behaved as spherical entities in solutions.

## CONCLUSIONS

A series of poly(azomethine ester) copolymers were synthesized with the solution polycondensation technique at about 25°C. In TGA and DSC data, all polymers showed good thermal stability, and no significant weight loss was observed until 370°C. The solubility study showed that PAM-2, PAM-4, and PAM-5 were soluble in NMP, DMF, PCP, and concentrated  $H_2SO_4$ . The substitution of a methoxy ( $-OCH_3$ ) group was useful for improving the solubility and for bringing down  $T_m$  and  $T_i$  temperatures. Except for PAM-3, all polymers showed nematic LC behavior. WAXD showed that all polymers were semicrystalline in nature and showed crystalline peaks around  $2\theta = 20^\circ$ . A major change was observed in the crystallinities of PAM-4 and PAM-1 near their isotropic temperatures 225°C and 300°C, respectively. They became amorphous at  $T_i$ . The solution viscosity studies showed a decrease in  $V_E$  and  $[\eta]$  with increase in temperature. No significant change was observed in solution viscosity due to the introduction of a methoxy group in the main-chain polymer backbone. The poly(azomethine esters) showed much lower  $[\eta]$ 's in a given solvent in comparison to some polyesters, as mentioned earlier. From all of the studies mentioned previously, we concluded that nature of the mesogenic group and the substitution of the group are very important factors that influence the solubility and thermal properties of the polymers.

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