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Light scattering from a nematic monodomain in an electric field

Twist elastic constant and viscosity coefficient of nematic polymer–solvent mixtures

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The light scattering technique was used to investigate the viscoelastic parameters characterizing director twist distortions in miscible nematic mixtures of 5CB (pentacyanobiphenyl) with two side chain liquid crystal polymers and a main chain liquid crystal polymer. By applying an AC electric field to homeotropically-aligned nematic monodomains of the mixtures, the field-dependent scattering intensities and director orientation fluctuation relaxation rates yield, respectively, the twist elastic constant K_{22} and viscosity coefficient γ_1. The results directly demonstrate that the addition of liquid crystal polymers causes substantial decreases of the relaxation rates for dynamic light scattering from the twist mode and these changes are due to small decreases in K_{22} coupled with large increases in γ_1. The decrements in K_{22} are comparable for both side chain and main chain liquid crystal polymers. The relative increase in the twist viscosity for the side chain liquid crystal polymers is much smaller than those of main chain polymers. A theoretical model is used to qualitatively interpret the difference between the viscous behaviour of the twist mode for both side chain and main chain liquid crystal polymers in a nematic solvent.

1. Introduction

The analysis of depolarized light scattered by thermally driven long wavelength fluctuations of the director orientation in a nematic monodomain is an effective method to characterize the viscoelastic properties of the nematic matrix [1]. The differential scattering cross-sections for two diffusive deformation modes, corresponding to splay–bend and twist–bend director distortions, are inversely proportional to the Frank elastic constants K_{ii} [1], where $i = 1, 2, 3$, refer, respectively, to the splay, twist, and bend distortions [1]. The relaxation rates of each mode are proportional to the ratios of the various elastic constants to viscosity functions. These can be determined by dynamic light scattering which involves photon correlation analysis of the scattering light [1–6]. By choosing appropriate combinations of scattering angle, polarization of the incoming and scattered light, and the director orientation, we can measure the scattering intensities and the relaxation rates of the pure splay, twist, and bend modes [7] and thus obtain ratios between elastic constants as well as ratios of the elastic constants to the viscosity coefficients. In order completely to characterize the viscoelastic behaviour of a nematic system, we need to obtain the individual values of the elastic constant and viscosity coefficients. Separation of the viscosity and elasticity

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contributions to the relaxation rates in light scattering experiments can be achieved by application of an external electric field. As discussed by de Gennes [1], the applied field decreases the amplitude of the director orientation fluctuations, making the scattering intensity and the relaxation rate field-dependent. In particular, if an electric field is applied along the director of a homeotropic monodomain which has a positive dielectric anisotropy, the depolarized scattering intensity and relaxation rate of the pure twist distortion become, respectively [1, 8],

$$\frac{d\sigma}{d\Omega}=\frac{A}{K_{22}q_\perp^2+\varepsilon_0\Delta\varepsilon E^2} \tag{1}$$

and

$$\Gamma_2=K_{22}q_\perp^2/\gamma_1+\varepsilon\Delta\varepsilon E^2/\gamma_1 \tag{2}$$

where ε_0 is the electric permittivity in vacuum, E is the field strength, $\Delta\varepsilon$ is the dielectric anisotropy of the liquid crystal, and A is a constant at certain temperature and scattering geometry. Therefore, the twist elastic constant and viscosity can be obtained directly by measuring respectively the field dependence of the scattering intensity and relaxation rate. This method was used by Leslie and Waters [8] and by Coles and Sefton [9] to obtain the twist elastic constant and the twist viscosity of 5CB and its mixtures with certain side chain liquid crystal polymers.

The viscoelastic behaviour of nematic monodomains containing polymers has been investigated by several groups using dynamic light scattering. One class of systems studied are lyotropic materials consisting of concentrated solutions of stiff chain polymers such as helical poly(*g*-benzylglutamate) (PBG) [10, 11], and poly(1,4-phenylene-2, 6-benzobisthiazole) (PBT) [12]. The relaxation rates for the splay and twist modes in such systems are much slower than those of low molar mass nematics, whereas the bend relaxation rates are near those typical of low molar mass nematics. A second class of systems studied comprise miscible mixtures of liquid crystal polymers dissolved in low molar mass nematogens [5, 7, 13–15]. The addition of a liquid crystal polymer causes a substantial decrease in the relaxation rates of the director distortion modes. For side chain liquid crystal polymers, it has been shown that this effect is largely a result of increases in the viscosity coefficients. Obviously, the configuration of a polymer chain in a nematic solvent will have a large influence on the viscoelastic behaviour of the monodomain. In a theoretical analysis of the viscosity increments due to addition of polymer chains into a nematic fluid, Brochard [16] developed expressions in terms of a few microscopic parameters such as $R_\perp$, $R_\parallel$ (the radii of gyration of polymer perpendicular and parallel to the director), and the rotational relaxation time of the chain. This theory suggests that information on the polymer chain configuration in a nematic solvent can be estimated by measuring the increments of the various viscosity coefficients. However, very few experimental attempts have been made to investigate the connection between polymer chain configuration in anisotropic solvent and viscosity measurements. In our recent studies [7, 17], we examined the relaxation rates of the splay, twist, and bend distortion modes via dynamic light scattering for side-chain liquid crystal polymer and main chain liquid crystal polymer mixtures with 5CB at various concentrations, molecular weights, and spacer lengths. We found: (*a*) for side chain liquid crystal polymers, the relative decrease of the relaxation rate of the bend mode was comparable to or larger than those of the splay and twist modes and strongly dependent on spacer length, backbone flexibility, and molecular weight; (*b*) for main chain liquid crystal polymers, in contrast, the relative decrease in the relaxation rates is much larger for the splay and twist modes than for the bend mode, and the change is enhanced by the increase of the chain contour length for splay and twist modes but not for the bend mode. Both (*a*) and (*b*) lead us to conclude that the interaction between the nematic field and the pendant mesogenic groups makes the mesogenic groups align with the nematic director, inducing the chain backbone to orient perpendicular to the director for side chain liquid crystal polymers and parallel to the director for main chain liquid crystal polymers. Here we report analysis of static and dynamic light scattering on nematic mixtures containing liquid crystal polymers with differing molecular architectures under an AC electric field. We are able to determine separately the twist elastic constant K_{22} and the twist viscosity γ_1 and demonstrate directly that the addition of a liquid crystal polymer to the nematic solvent results in small decreases of the elastic constants, and large increases of the viscosities. Our results confirm the earlier deduction that the decrease of the relaxation rates is due principally to increase of viscosity and provide further evidence that the anisotropy in the dynamical behaviour of the director distortion modes is a consequence of the fact that the interaction between the nematic field and the mesogenic groups induces distinct asymmetric chain configurations for main chain and side chain liquid crystal polymers.

2. Experimental

The low molar mass nematic liquid crystal solvent 5CB used in this study was purchased from BDH Ltd and used as received. Figure 1 shows the chemical structures of the three liquid crystal polymer species used in our study. Two are side chain liquid crystal polymers: poly[6-[(4-methoxy-α-methylstilbene-4-yl)oxy]hexyl methacrylate] (MSHMA) and poly[ω-[4-cyano-4′-biphenyl)oxy] septyl vinylether](7-PVE). The third is a main chain liquid crystal polymer which consists of the mesogenic group, 1-(4-hydroxy-4′-biphenyl)-2-(4-hydroxy phenyl)butane, separated by flexible spacers of variable length, $n=5,7,9,11$ (TPB-*n* polyethers). Each of these polymers has been extensively characterized [18–20]. At low concentrations, each liquid crystal polymer was found miscible with 5CB in both nematic and isotropic states. A Carl Zeiss optical polarizing microscope equipped with a Mettler FP 82 hot stage and a Mettler FP 800 central processor was used to evaluate the miscibility and determine the nematic to isotropic transition temperature T_{NI} of the sample. The T_{NI} of 5CB was measured as 35·0°C. The T_{NI} for the liquid crystal polymer mixtures were found to be slightly higher (0·2 ~ 2°C) than that of 5CB and to exhibit a narrow biphasic region at the T_{NI}, depending on the polymer concentration. The nematic mixtures were sandwiched between two clean, conductively-coated glass slides separated by 25 μm Mylar spacers. The thickness d of the sample cell was measured using an interferometric technique [21] with an experimental error of 1 per cent. The planar and homeotropic monodomains were prepared using surface treatment techniques described elsewhere [7]. The sample cells were filled by capillary action and sealed with epoxy (DEVCON).

The dielectric anisotropy $\Delta\varepsilon=\varepsilon_{\parallel}-\varepsilon_{\perp}$ is obtained by measuring the capacitance of the sample cell while an increasing bias voltage is applied to the planar aligned monodomain cell [22]. The frequencies of the bias voltage and the probe signal were 50 Hz and 1000 Hz, respectively. The field-dependent dielectric constant ε was determined by comparing the measured capacitance and the empty cell capacitance which was measured before the cell was filled. The sample cell was placed in an oven whose temperature was regulated 5°C below the T_{NI} of the nematic mixture by a temperature controller (YSI model 72) accurate to 0·2°C. The value of $\varepsilon_{\perp}$ was measured

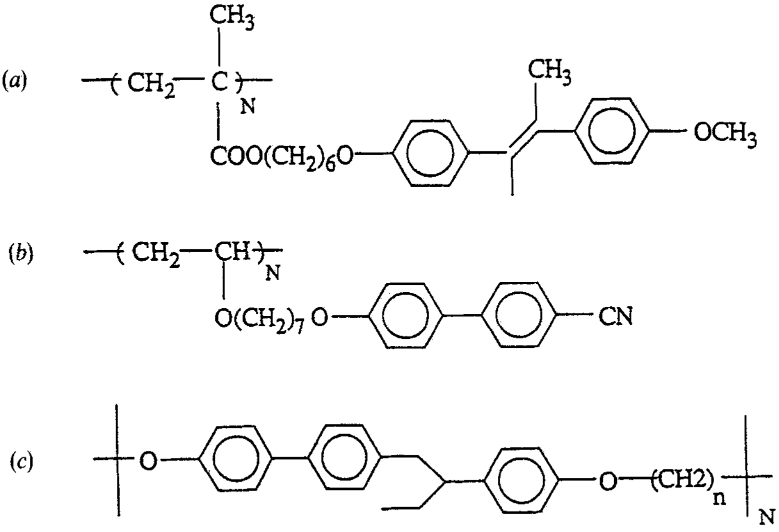

Figure 1. Molecular structures of the liquid crystal polymers studied in this investigation: (*a*) MSHMA, (*b*) 7-PVE, (*c*) TPB-*n*-polyether.

at zero field and the value of $\varepsilon_{\parallel}$ was determined by extrapolating the $\varepsilon - 1/V_b$ curve to infinite voltage [22]. The overall accuracy in measuring $\Delta\varepsilon$ is about 3 per cent.

Light scattering measurements were performed in the homodyne configuration using a photon correlation spectrometer equipped with a 15 mW He/Ne laser and a BI 2030 AT 256-channel digital correlator. The sample cell containing homeotropically aligned monodomain was positioned in a refractive index-matching bath containing toluene using a microtranslator which enables us to locate the scattering volume in the focal plane of the collecting lens. The incident laser beam is normal to the sample surface so that the director is parallel to the incident wave vector. The polarization of the incident light is perpendicular to the scattering plane and the scattered light is detected with its polarization in the scattering plane. At relatively small scattering angles in this scattering geometry, the scattering is principally due to the twist mode with a minor contribution from the bend mode because of a very large ratio of $q_{\perp}^2/q_{\parallel}^2$. For example, at scattering angle of 18° in lab frame, $q_{\perp}^2/q_{\parallel}^2 = 63$, and hence the contribution from the bend mode to the relaxation rate Γ_2 is comparable to experimental error (about 2 per cent). The refractive indices for 5CB and the mixtures were determined using ABBE refractometer model 60/HR equipped with a Fisher refrigerated circulator, model 9100. The sample temperature was controlled, in each case, at 5°C below the T_{NI} of the nematic mixture by a refrigerated circulating bath, accurate to better than 0·1°C. The scattering intensity autocorrelation functions obtained by photon correlation analysis exhibit single exponential decay within measurement error, confirming that the pure twist mode has been detected. The AC electric field applied to the homeotropic sample was provided by an audio-frequency signal generator (HP model 200CDR). When field frequencies were lower than 1000 Hz, a sinusoid signal with frequency at twice the field frequency was found superposed on the measured autocorrelation function and the amplitude of the sinusoid signal was inversely proportional to the square of field frequency. This superposed signal presumably occurs when the director relaxation rate becomes comparable to or higher than the field frequency so that the scattering intensity can follow the electric field to go on a periodic change. Observation of the more concentrated nematic mixtures confirms that, when the viscosity of the system is increased, which means a lower director relaxation rate, the amplitude of the modulated structure diminishes. To avoid complications in data analysis, a frequency of 3000 Hz was selected for the homodyne detection of the scattered light in our experiment. At this frequency, within the noise level (0·3 per cent), no sinusoid signal was observed on the autocorrelation functions for any of our samples.

3. Results and discussion

As a direct demonstration of our earlier conclusion that the addition of liquid crystal polymer to a low molar-mass nematic matrix causes a small decrease in the twist elastic constant and a large increase in the twist viscosity, we exhibit field-dependent scattering intensity and relaxation rate for mixtures of the main chain liquid crystal polymer TPB-11, at concentrations $0 \sim 3\%$ w/w, and for two side chain liquid crystal polymer mixtures at 8% w/w concentration. In figure 2, the inverse relative scattering intensities of 5CB and the mixtures of main chain liquid crystal polymer TPB-11 are plotted against the field parameter $\varepsilon_0 \Delta\varepsilon v^2/d^2$. From equation (1), the slopes of these least square linear fits to the experimental data yield the inverse twist elastic constants K_{22}. Compared to 5CB, it can be seen from these intensity measurements that the change in K_{22} due to addition of liquid crystal polymer is rather small. Specifically, differences between the values of K_{22} for 5CB and the liquid crystal polymer mixtures are comparable to the experimental error, which is about 10 per cent. In figure 3 the corresponding field-dependent relaxation rates of the twist mode for TPB-11 mixtures are shown. From equation (2) the slopes of the linear fits yield the inverse twist viscosities. Two features of the linear fits in figure 3 are of particular note: (*a*) the intercepts which correspond to zero-field relaxation rates, and the slopes decrease with increase of liquid crystal polymer concentration, (*b*) the relative decrements of both the zero-field relaxation rates and the slopes are linearly proportional to the polymer concentration. Since the former are ratios of the product of twist elastic constant and the scattering vector to the twist viscosity, while the latter are the inverse twist viscosities, we can conclude again that the twist viscosity is dominant in these changes. The linearity of the decrements with respect to polymer concentration is evidence that the polymers are in the dilute solution regime rather than in an overlapping or entanglement regime where the viscosity would show a power law behaviour. Comparing the precision of the results in figures 2 and 3, we find a more accurate measurement of K_{22} can in fact be achieved from the field dependent relaxation rates, using γ_1 determined from the slopes of the linear fits. In the table, we summarize the experimental results for the main chain and side chain liquid crystal polymer mixtures utilized in this study.

One approach to investigate the effect of the nematic field on chain configuration is to examine the chain length dependence of the viscosity increment or the intrinsic viscosity. For the intrinsic twist viscosity $[\gamma_1]$,

$$[\gamma_1] = (\gamma_1 - \gamma_1^0)/\gamma_1^0 C, \tag{3}$$

where γ_1^0 is the twist viscosity of pure 5CB and C is the mass concentration. In figure 4 the intrinsic twist viscosities for the main chain liquid crystal polymer samples with different molecular weights and spacer lengths are plotted against contour length

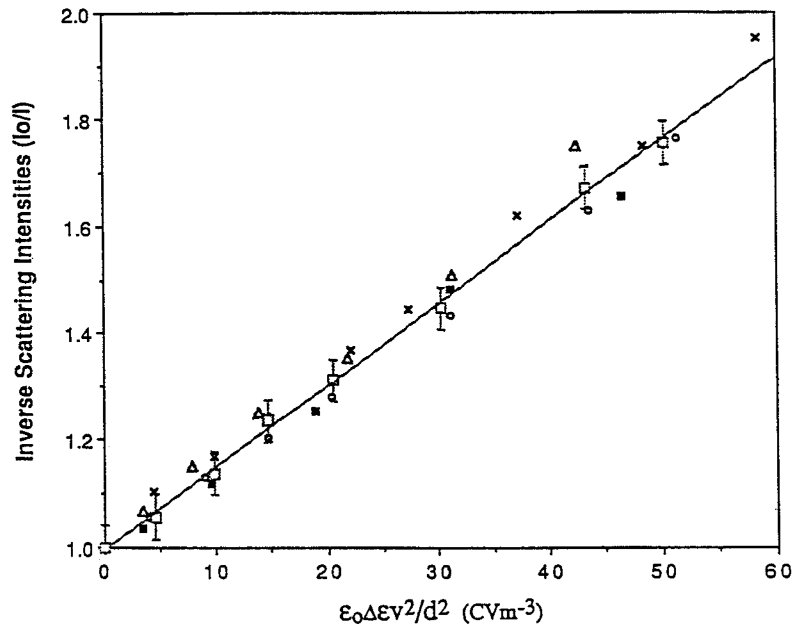

Figure 2. Electric field dependence of the inverse scattering intensities for the main chain (TBP-11)/5CB mixture at various concentrations: (□) pure 5CB; (○) 0·5% w/w; (×) 1·0%w/w; (■) 1·5% w/w; (△) 3·0% w/w. The inverse slopes of the lines in this figure are $K_{22}^{2}q_{\perp}^{2}$, from which the twist elastic constant can be determined. Typical error bars are shown.

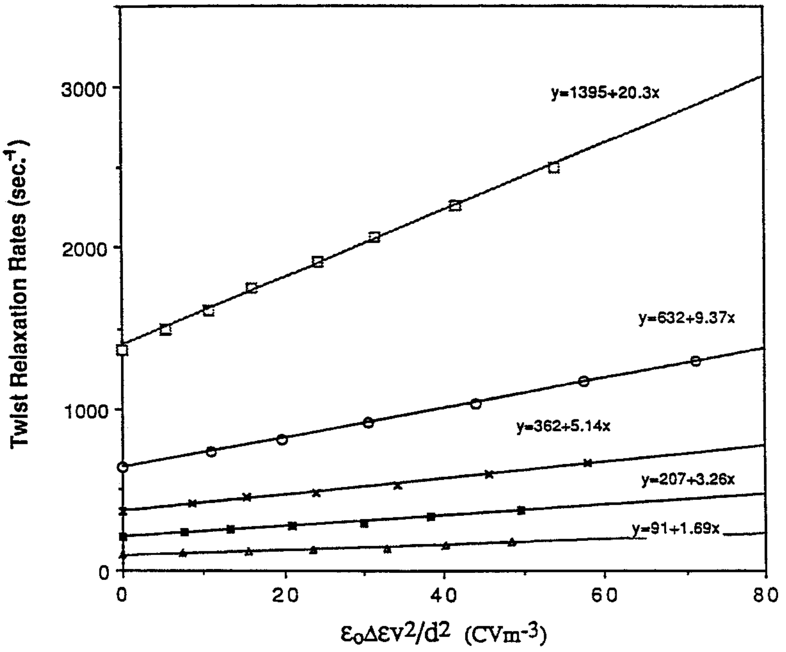

Figure 3. Electric field dependence of the director relaxation rates for the twist mode at different concentrations for the main chain liquid crystal polymer (TPB-11)/5CB mixture. Symbols used here are as in figure 2. The slopes of the lines in this figure are the inverse of the twist viscosity γ_1 for the nematic mixtures.

L(nm) of the fully-extended chain, the latter computed from the measured molecular weights. Least squares fit to the data in figure 4 leads to a relation

$$[\gamma_1] = 0{\cdot}90L(\text{nm})^{1{\cdot}26} \tag{4}$$

Compared with our exponent of 1·76 for the intrinsic relaxation rate decrement [7], the viscosity exponent 1·26 is smaller, the difference reflecting the small decrease in twist elastic constants which is also a consequence of addition of polymer. In Brochard's theoretical analysis [16] of the viscosities for nematic mixtures, the increment of the twist viscosity due to presence of polymer can be expressed as

$$\delta\gamma_1 = Ck_BT(R_\perp^2 - R_\parallel^2)^2\tau_R/(R_\perp^2 R_\parallel^2 N), \tag{5}$$

Viscoelastic constants of twist mode for polymer mixtures.

Sample	DP (N)†	$K_{22}/10^{-13}$N	γ_1/P	$[\gamma_1]$/(g/g)
Pure 5CB	—	32	0·49	—
8% MSHMA	29	29	1·05	14
8% 7-PVE	30	30	0·72	5·9
3% TPB-5	42	30	4·4	260
3% TPB-7	37	32	4·2	250
3% TPB-9	60	30	8·6	550
3% TPB-11	44	30	5·8	370

† DP is degree of polymerization.

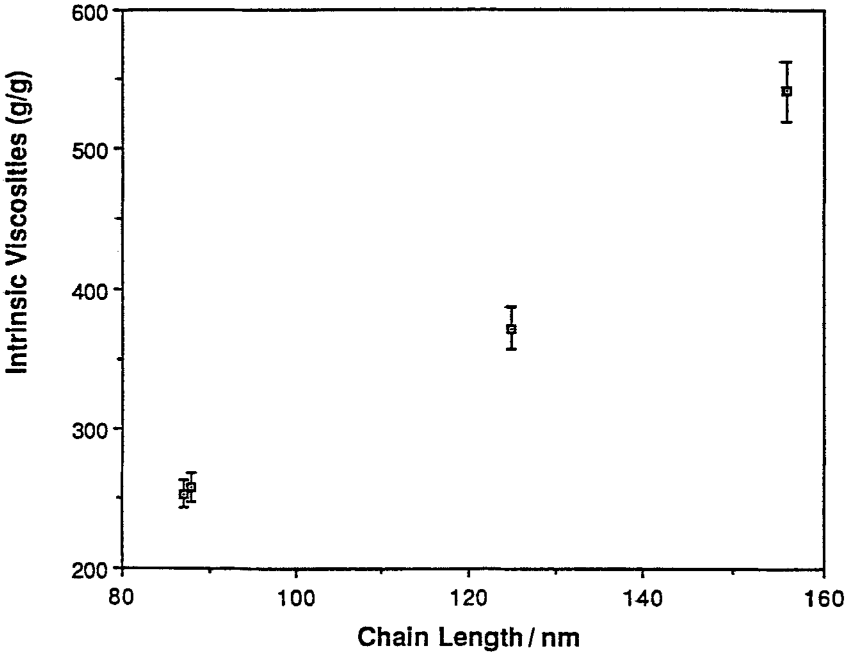

Figure 4. The intrinsic twist viscosities for the nematic mixtures of main chain liquid crystal polymer (TPB-5, 7, 9, 11) with 5CB, plotted as function of chain contour length (nm).

where N is degree of polymerization, k_B is Boltzmann constant, T is temperature, $R_{\|}$ and $R_{\perp}$ are the radii of gyration parallel and perpendicular to the director respectively, and τ_R is the rotational relaxation time of the polymer chain

$$\tau_R = \lambda_{\perp}\lambda_{\|}R_{\perp}^2R_{\|}^2/(\lambda_{\perp}R_{\perp}^2 + \lambda_{\|}R_{\|}^2)KT, \tag{6}$$

where $\lambda_{\|}$ and $\lambda_{\perp}$ are the frictional coefficients for the translational chain motion parallel and perpendicular to the director. Equation (5) indicates that if $\delta\gamma_1 \neq 0$, then a significant anisotropy in chain configuration must exist. Assuming that a main chain liquid crystal polymer will tend to align with the director, $R_{\|} \gg R_{\perp}$, and equation (5) deduces to

$$\delta\gamma_1 = Ck_BT\lambda_{\perp}R_{\|}^2/N. \tag{7}$$

Assuming a fully-extended chain, for which $\lambda_{\perp} \propto N$, and $R_{\|} \propto N$, we obtain

$$[\gamma_1] \propto N^2. \tag{8a}$$

Alternatively, for a one dimensional random walk

$$[\gamma_1] \propto N^{0\cdot 5}. \tag{8b}$$

The experimental exponent 1·26 is intermediate between the two extreme values in equation (8) suggesting that the chain is intermediate between the random and fully-extended states.

In figures 5 and 6, the field dependent scattering intensities and the twist relaxation rates for two side chain liquid crystal polymer mixtures MSHMA and 7-PVE with similar molecular weights and concentration are plotted and compared with pure 5CB. Again we see directly that the addition of polymer causes little change in K_{22} and large

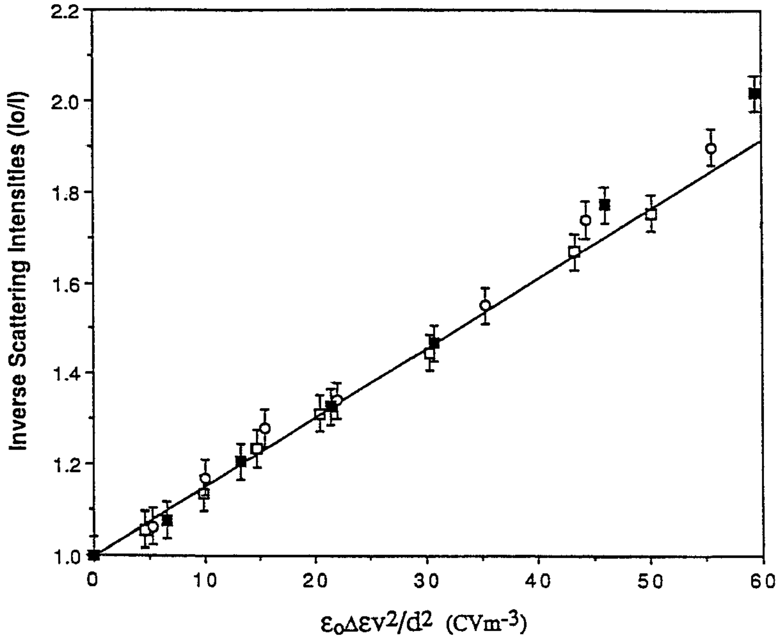

Figure 5. Electric field dependence of the inverse scattering intensities for the side chain liquid crystal polymer MSHMA and 7-PVE in 5CB, compared with that of pure 5CB: (■) 8% w/w MSHMA; (○) 8% w/w 7-PVE; (□) pure 5CB.

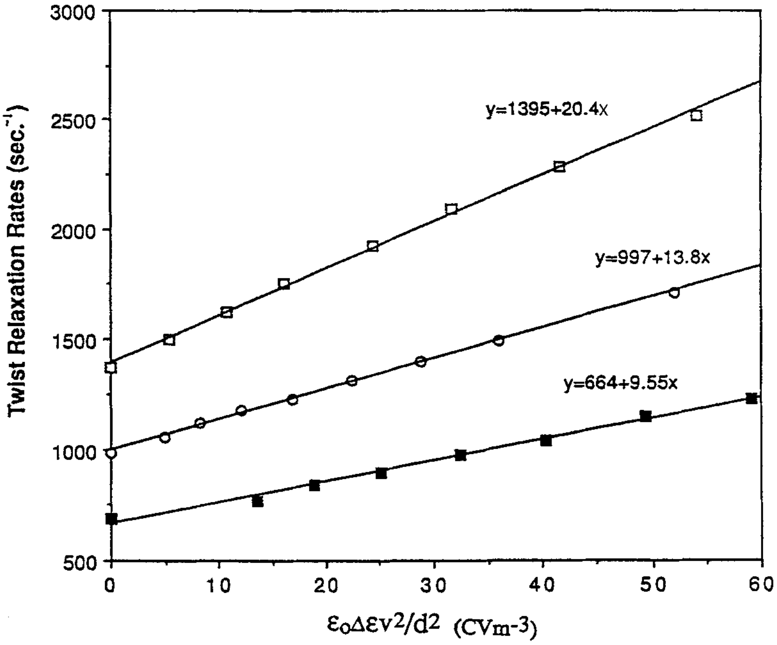

Figure 6. Electric field dependence of the director relaxation rates for the twist mode for the side chain liquid crystal polymer MSHMA and 7-PVE in 5CB, compared with that of pure 5CB. Symbols used are as in figure 5.

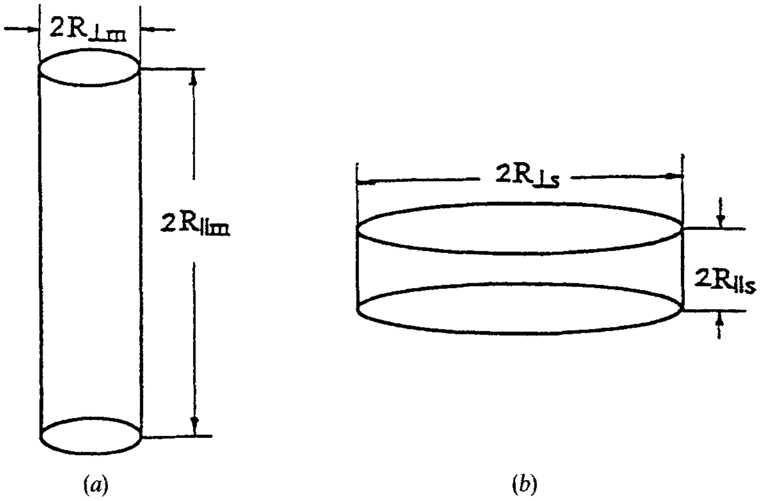

Figure 7. Geometric illustration for the configurations of (*a*) main liquid crystal polymer and (*b*) side chain liquid crystal polymer in nematic solvent. For simplification, $R_{\parallel s} = R_{\perp m}$ is assumed for polymers with similar molecular weight.

changes in γ_1. It is also evident that even at higher concentration 8% w/w, the increase of the twist viscosity for each side chain liquid crystal polymer is smaller than that of the main chain liquid crystal polymer mixtures at concentration of 3% w/w by nearly an order of magnitude. Again, the substantial magnitude of $\delta\gamma_1$ observed for side chain liquid crystal polymers implies via the Brochard theory that the chain configuration is anisotropic ($R_\parallel \neq R_\perp$). For the limit $R_\parallel \ll R_\perp$, equation (5) reduces to

$$\delta\gamma_1 = Ck_B T\lambda_\parallel R_\perp^2/N. \tag{9}$$

Note that, if we assume the frictional coefficients $\lambda_\perp$ and $\lambda_\parallel$ in equations (7) and (9) can be expressed in terms of solvent viscosity γ_1^0 and two apparent Stokes hydrodynamic radii, $\lambda_\perp = 6\pi\gamma_1^0 R_m^H$ and $\lambda_\parallel = 6\pi\gamma_1^0 R_s^H$, where m and s refer to side chain and main chain LCP respectively, $R_m^H = \xi R_{\parallel m}$ and $R_s^H = \xi R_{\perp s}$where ξ is a parameter which describes the strength of hydrodynamic interactions, then taking the ratio of equations (7) to equation (9), we have

$$R_{\parallel m}^3/R_{\perp s}^3 = \delta\gamma_{1m}N_m C_s/\delta\gamma_{1s}N_s C_m = [\gamma_1]_m N_m/[\gamma_1]_s N_s. \tag{10}$$

Comparing equation (10) with our experimental results for TPB-7 ($N=37$) and MSHMA ($N=29$), we find $R_{\parallel m}^3/R_{\perp s}^3 = 16$. Thus this large ratio of the intrinsic twist viscosity for main chain and side chain liquid crystal polymers with comparable molecular weights is consistant with the idea that the chain configuration for main chain and side chain liquid crystals polymers in a nematic solvent are, respectively, prolate and oblate ellipsoids. In fact, simple geometric arguments indicate that, for two cylinders of equal volume and equal minimum dimension but different shapes as shown in figure 7, the experimental observation $R_{\parallel m}^3/R_{\perp s}^3 = 16$ implies $R_{\perp s}/R_{\parallel s} = 2{\cdot}5$, and $R_{\parallel m}/R_{\perp m} = 6{\cdot}3$. If free-draining is assumed for both the main chain and side chain liquid crystal polymer mixtures, we have $\lambda_{\parallel s} \propto N_s$ and $\lambda_{\perp m} \propto N_m$, then the ratio of equation (7) to equation (9) becomes

$$R_{\parallel n}^2/R_{\perp s}^2 = [\gamma_1]_m/[\gamma_1]_s \tag{11}$$

suggesting that $R_{\perp s}/R_{\parallel s} = 4{\cdot}5$, and $R_{\parallel m}/R_{\perp m} = 20$ in our experiments. Such values appear to be quite reasonable, based on small angle neutron scattering studies of deuterated main chain [23] and side chain liquid crystal polymer [24–26].

According to figure 6 and the table, the increment in twist viscosity of the 7-PVE mixture is about 45 per cent smaller than that of the MSHMA mixture, despite the similarity of their molecular weights, indicating the different chemical structures in these two polymers cause the viscosity difference. The pendant methyl group vicinal to the mesogenic side chain in MSHMA reduces the flexibility of the polymer backbone. In addition, in contrast to the ether linkage of 7-PVE, MSHMA has an ester linkage between the side chain and backbone which makes the spacer less flexible and the mesogen motion less decoupled from the backbone. Each of these factors tends to increase the anisotropy in chain backbone configuration in MSHMA relative to 7-PVE, inducing larger twist viscosity increments (cf. equation (5)). Currently, we are attempting to compare the molecular weight dependence of the viscosity coefficients in MSHMA and *n*-PVE to confirm this.

4. Conclusions

We have investigated the light scattering of twist distortion modes of the director under an applied electric field for nematic mixtures of low molar mass liquid crystal and liquid crystal polymers with different architectures. The results confirm that in the presence of an applied field, dynamic light scattering is an effective technique to obtain individual values of both viscosity coefficients and elastic constants. The slowing down of the twist relaxation rates of director fluctuation in 5CB on addition of main chain liquid crystal polymer and side chain liquid crystal polymer was observed and this change is due to a large increase in the twist viscosity and a minor decrease of the twist elastic constant. The change in the viscosities for the twist mode of main chain liquid crystal polymer mixtures is much larger than that of the side chain liquid crystal polymer mixtures. This anisotropic behaviour in the twist viscosity increment can be explained by the idea that the nematic field orients the polymer chain backbone parallel to the director for the main chain liquid crystal polymer, producing a cylindrical configuration, while for the side chain liquid crystal polymer, the mesogenic groups are aligned perpendicular to the chain backbone, resulting in a disc-like configuration.

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