Proton field-cycling nuclear magnetic resonance relaxometry in the smectic A mesophase of thermotropic cyanobiphenyls: Effects of sonication

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Proton field-cycling nuclear magnetic resonance relaxometry is used to study the spin-lattice relaxation dispersion of selected standard smectic A liquid crystals at different temperatures. Relaxation features at both, in the presence and absence of a monochromatic ultrasonic field are considered. We show that the laboratory-frame spin-lattice relaxation time is mainly governed by translational diffusion. Order director fluctuations (ODF) are less important while rotational diffusion seems to be only relevant near the clearing point. Our study suggests that sonication enhances the ODF contribution in the SmA mesophase. Within the framework of the approach we have outlined, different features associated with the ODF mechanism can be investigated. © 2004 American Institute of Physics. [DOI: 10.1063/1.1740751]

I. INTRODUCTION

The Larmor frequency $\nu$ dispersion of proton spin-lattice relaxation time $T_1$ in thermotropic liquid crystals may be analyzed through the superposition of different competing relaxation mechanisms. Among the most relevant we may consider collective molecular motions like order director fluctuations (ODF), and individual molecular motions such as translational and rotational self-diffusions. It should be emphasized that the smectic A (SmA) mesophase was scarcely studied with the field-cycling technique. Remarkably, some features related to the $T_1$ relaxometry of these systems are still left as an open question. A characteristic of these systems is the strong dispersion of the $T_1$ appearing in the low-frequency end of the available Larmor frequency window. Such strong dispersion was usually associated with the smectic ODF mechanism. However, it was recently shown that, at least for cyanobiphenyls, such dispersion is more connected with hardware setting and the presence of local fields. Since we have lost the ideal scenario of a $T_1$ relaxation parameter measurable within the local field region, we decided to investigate again the SmA mesophase with the main spirit of disentangling ODF and diffusion contributions.

Two main theoretical models have been proposed up to now for the SmA mesophase. The former, predicts a linear behavior of $T_1$ with Larmor frequency $\nu$ [\(T_1(\nu) = a\nu\)]. However, this Larmor frequency dependence was estimated from the undulation layer mechanism in the limit where the correlation length $\xi$ does not depend on the wave vector of the hydrodynamic modes $q$. If a more realistic relationship $\xi \approx 1/q^2$ is assumed, a logarithmic dependence for $T_1(\nu)$ is obtained. A second relaxation model proposed by Vilfan et al. foresees a behavior for $T_1(\nu)$ ranging approximately from $\nu^0$ at low frequencies (minor than 1 MHz) up to $\nu^{1.5}$ in the high frequency regime (in logarithmic scale). In a limiting case (when the SmA phase only involves splay deformations and compressions of the layers), the above mentioned logarithmic behavior is reobtained.

The characteristic leveling-off behavior predicted by this last model at low frequencies anticipates the difficulties that may arise in disentangling ODF from diffusion or other non-collective mechanisms. The situation can be partially circumvented by extending the relaxation dispersion study to higher frequencies, outside the technical limits of the field-cycling method (by measuring $T_1$ at fixed frequencies using standard pulse sequences). But a final word depends on how strong can be the dominance of individual molecular motions over the collective ones within that extended high frequency range. This last comment warrants a remarkable observation: There is no reason a priori to assume that collective motions are slow, since they may be effective up to frequencies corresponding to the high frequency cutoff (associated with molecular dimensions) within the high MHz regime. A completely different point is that ODF dominates, for example, the relaxation dispersion of nematogens in the kHz region.

The typical dispersion of the $T_1$ relaxation time experimentally observed in the SmA phase of cyanobiphenyls is characterized by a weak frequency dependence between 1 MHz and a frequency of about 20 kHz. At this frequency a noticeable steep dispersion appears until about 800 Hz, where a low frequency plateau manifests. The strong dispersion was usually explained through order director fluctuations, while the low frequency plateau was interpreted in terms of the low-frequency ODF cut-off mechanism. In most of these works, the simplified model that predicts a linear ODF dispersion was invoked. However, recent studies suggest that the observed steep dispersions are a conse-
NMR relaxometry in the smectic A mesophase

II. EXPERIMENT

Commercial samples of 4-octyl-4'-cyanobiphenyl (8CB) and 4-undecyl-4'-cyanobiphenyl (11CB) from Synthon and Merck Chemicals, respectively, were employed. 8CB presents three phase transition: Solid (K)–SmA at 294.5 K, SmA–N at 306.5 K and N–isotropic (I) at 313.5 K, while 11CB only displays two phase transitions: K–SmA and SmA–I at 326 and 330.5 K, respectively.

A Stelar FC2000 fast field cycling nuclear magnetic resonance (NMR) relaxometer was used to acquire all the proton field-cycling NMR triplet of the chain groups. Self diffusion was found to be the more important mechanism driving the $T_1$ relaxation in the SmA phase. This assertion was generally checked to be consistent with our results. Therefore, in the following we will refer to self diffusion as the only important individual relaxation mechanism.

III. RELAXATION MODELS

As it was previously stated, three different relaxation mechanisms were used to explain the $T_1(\nu)$ behavior: One of collective nature (ODF) and two of individual nature (translational diffusion and molecular reorientations). By translational diffusion mechanism, or self diffusion, we mean the modulation of the dipole–dipole interaction between protons located on different molecules, as a consequence of the particle motion, usually thermally activated, from one site to another. Reorientations are intended to be the contributions to the spectral densities at the Larmor frequency caused by rotations around the long and short molecular axis, and rotations of the chain groups. Self diffusion was found to be the more important mechanism driving the $T_1$ relaxation in the SmA phase. This assertion was generally checked to be consistent with our results. Therefore, in the following we will refer to self diffusion as the only important individual relaxation mechanism.

Assuming different time scales for the different mechanisms and small cross terms between them at the correlation function level,

\[
\frac{1}{T_1} = \frac{a_1}{T_{1\text{OF}}} + \frac{a_2}{T_{1\text{Tr}}},
\]

where $T_1^{-1}$ is the total proton spin relaxation rate; $a_1$ and $a_2$ are constants; and $T_{1\text{OF}}$, $T_{1\text{Tr}}$, are the relaxation rates produced by ODF and translational diffusion mechanisms, respectively, in the absence of the other mutually relaxation mechanisms.

At this point, it is important to remark that due to the low spectroscopic resolution of the field-cycling experiments, separate contributions from phenyl and chain protons, at least for these compounds, are not distinguishable. Rapid spin diffusion ensures a single average $T_1$, as can be concluded from the mono-exponential evolution of the magnetization decay.

As described in the preceding section, there are essentially two proposed models that may be considered to explain the Larmor frequency dispersion due to the ODF relaxation mechanism in the SmA phase:

- The “nematic” approach.

Consider the nematic free energy:

\[
\Delta F = \frac{1}{2} \int \frac{4\pi}{3} \rho \left( \frac{\text{d} \mathbf{u} \cdot \mathbf{r}}{\rho} \right)^2 \tau \text{d}^3 \mathbf{r},
\]

where $\rho$ is the density of the nematic director field, $\mathbf{u}$ is the director field, $\tau$ is the relaxation time, and $\mathbf{r}$ is the position vector.
where $K_{11}$, $K_{22}$, and $K_{33}$ are splay, twist and bend elastic constants, respectively, and $\vec{n}$ is the local director.

If the contribution of smectic undulation waves is taken into account while assuming that the correlation length in the direction perpendicular to the smectic layers does not rely on the mode's wave vector, we have:

$$T_{1\Omega F}^{se}(\nu) = C\nu,$$

where constant $C$ depends on the Boltzmann and Planck constants, the gyromagnetic ratio, temperature $T$, splay elastic constant $K_{11}$, the interproton distance, order parameter $S$, and the coherence length in the direction perpendicular to the smectic layers. The previous supposition is mathematically (but not physically) equivalent to assume that the relation $K_{33} \ll K_{11}, K_{22}$ is satisfied in Eq. (2).  

* The “smectic” approach.

In this model the coupling of smectic order with director fluctuations is taken into account.  

$$f = f_n + f_s + f_1,$$

with,

$$f_s = \epsilon(T)|\psi|^2 + \lambda(T)|\psi|^4 + \ldots$$

and

$$f_1 = (\nabla + iq_0\vec{d}\eta)\psi^* \frac{1}{2M}(\nabla - iq_0\vec{d}\eta)\psi.$$

In the last expressions, $\epsilon$ and $\lambda$ are coefficients in the expansion of $f_s$ in powers of $\psi$, $M$ is a mass tensor with components $M_i$ and $M_{ij}$, along the normal to the layers and perpendicular to them, respectively and $q_0 = 2\pi/d$, $d$ the distance between smectic layers and $\psi$ the smectic order parameter $[\psi = |\psi| \exp(i\phi)].$

Using the expression obtained in Refs. 17 and 18 for the amplitudes of the mean square fluctuations of the director for the $q$th mode ($\langle |n_a(q)|^2 \rangle$; with $a = 1, 2$) and their corresponding decay times $[\tau_{s}(q)]$, it is possible to arrive at

$$T_{1\Omega F}^{sm}(\nu) = \beta \left[ \frac{1}{\sqrt{\delta}} \eta_1 K_{11}^{-3/2} \right]$$

$$+ \frac{1}{\sqrt{K_{33} \delta}} \eta_2 K_{22}^{-1} \left( \frac{2\pi\nu}{\omega_{s2}} \right)^{-1},$$

with

$$Y = \frac{B}{\delta} \int_0^1 dx \left( \frac{B}{2} (1-x^2) + \frac{K_{33}}{K_{11}} x^2 \right)^{3/2} \sqrt{1 + \frac{2\pi\nu}{\omega_{s2}}},$$

where

$$B = \frac{\psi_0^2 q_0^2}{M}$$

$$\text{and} \quad \delta = \frac{\psi_0^2 q_0^2}{M}$$

($\psi_0$ is the equilibrium value of the smectic order), $\eta_a$ are viscosities, $\omega_{s2} = \delta |\eta_a|$, with $\alpha = 1, 2$, and $\beta$ depends on the Planck constant, the gyromagnetic ratio, the interproton distance and molecular order parameter $S$.

Concerning $T_{1\Omega}^{\tau^{-1}}$, relaxation rate, Vilfan et al. extended to SmA phases the model previously proposed by Torrey for the I phase. Although Vilfan et al. showed that for low frequencies the anisotropy of the molecules influences the relaxation rate, differences with Torrey’s model in the dispersion behavior become important only when the Larmor frequency $\nu$ varies around $2\pi \nu \tau_D < 0.3$, where $\tau_D$ is the diffusional correlation time. As in our experiments the factor $2\pi \nu \tau_D$ ranges from about $6 \times 10^{-5}$ to $2 \times 10^{-2}$ (only 7% of the range where both models differ significantly), in this frequency window we can approximate

$$T_{1\Omega}^{\tau^{-1}} = \chi T_{1\Omega sm}^{\tau^{-1}} (\nu),$$

with an appropriate factor $\chi$. In the last equation, $T_{1\Omega}^{\tau^{-1}} (\nu)$ is the expression obtained by Torrey for the I phase and $T_{1\Omega sm}^{\tau^{-1}} (\nu)$ is the expression obtained by Vilfan and Zumer for SmA phases.

Then, due to the exposed above and for the sake of simplicity, the following equation was used for $T_{1\Omega}^{\tau^{-1}}$:

$$T_{1\Omega}^{-1} = \frac{A_D}{\nu^2} \left[ j(\alpha, x) + j(\alpha, \nu^{2} x) \right],$$

where

$$j(\alpha, x) = u \left( 1 - \frac{1}{u^2 + v^2} \right) + \left( 1 + \frac{1}{u^2 + v^2} \right) + 2 e^{-2u} \times \cos(2u) + u \left( 1 - \frac{1}{u^2 + v^2} \right) \sin(2u),$$

$$\left( \frac{u}{v} \right) = \frac{1}{2} \left( \frac{q(1+q)}{\alpha} \right)^{1/2},$$

$$q = \frac{\alpha x^2}{(1 + \alpha x^2)^{1/2}}.$$
As it can be observed, this result no longer depends on $\alpha$. Although hardly justified, this approximated model was broadly used in the analysis of field cycling dispersion curves in liquid crystals in the past.\textsuperscript{4–10} In the present work, the above simplified model was checked to be inappropriate because of the $\tau_D$’s obtained by the corresponding fittings do not satisfy the hypothesis of low correlation times ($\omega \tau_D \ll 1$) assumed by the model in the whole $\omega$ spectrum.

A. Relaxation in the presence of ultrasound

Ultrasound couples with ODF causing changes in the whole spectrum of the relaxation dispersion curve.\textsuperscript{12,13} In this case, a new orienting term is added to the nematic free energy expression [Eq. (2)]. This term is given by\textsuperscript{12,21}

$$f_a = \frac{a^2}{2} \langle \delta \cdot \hat{n} \rangle,$$

where $a^2$ depends on the acoustic intensity, the ultrasound velocity, the average of the sample density, the magnitude of the ultrasound wave vector and the director-density coupling. In the last equation, $\delta$ is an unitary vector pointing along the ultrasonic wave vector, and $\hat{n}$ is the director.

Starting from this new free energy expression, the following is obtained for both, nematic and smectic approaches, in the presence of ultrasonic waves parallel to the director [the detailed calculation is offered in Ref. 14]:

$$T_{1OF+S}^{nem}(\nu) = \frac{3K_BT}{4\pi^2\omega K_{11}} \left\{ \frac{1}{\sqrt{1 + \left(\frac{1}{(\omega \tau_0)^2}\right)}} \times \left[ \frac{1}{\omega \tau_0} \arctan \left( \frac{1}{\sqrt{1 + \left(\frac{1}{(\omega \tau_0)^2}\right)}} + \pi \right) \right] \right\}^{-1},$$

$$T_{1OF+S}^{sm}(\nu) = \beta \left[ \frac{1}{\sqrt{D}} \eta_1 K_{11}^{-3/2} Y + j_{1,2}^{OF+S}(\omega) \right]^{-1},$$

with
\[
Y = \frac{B}{D} \int_0^1 \frac{dx}{\left(\frac{B}{D} \left(1-x^2\right) + \frac{K_{33}}{K_{11}} x^2\right)^{3/2}} \sqrt{x^2 - \frac{a^2}{D} + \sqrt{x^2 - \frac{a^2}{D}^2 + \left(\frac{\omega}{\omega_1}\right)^2}}.
\]

(15)

\[
J_{1/2}^{D_F+S} (\omega) = \begin{cases} 
\frac{1}{\sqrt{1+\left(\frac{\omega}{\omega_2}\right)^2}} & \text{if } D < a^2 \\
\frac{1}{\sqrt{1+\left(\frac{\omega}{\omega_2}\right)^2}} & \text{if } D \geq a^2 
\end{cases}
\]

(16)

In Eqs. (13)–(16), \( \tau_0 = \eta/a^2 \), \( \omega_2 = |D - a^2|/\eta_2 \) and \( \omega_{11} = D/\eta_1 \).

Based on experimental facts,\(^{12,13}\) it will be assumed that translational self-diffusion relaxation mechanism is not affected by the presence of ultrasonic waves.

**IV. RESULTS**

Figures 1 and 2 show the \( T_1 \) dispersion plots corresponding to a sample of 8CB at temperatures of 295 and 304 K within the SmA phase, respectively. Figures 1(a) and 2(a)
show the data obtained in the absence of ultrasonic waves (filled circles) and in the presence of them: At a power of 13.5 W/cm² (hollow stars) and at a power of 22.5 W/cm² (hollow circles). Figures 1(b) and 2(b) show the corresponding simulated curves for the nonsonicated case (solid line) and in the presence of ultrasonic waves (dashed and dotted lines). Figures 3 and 4 show the corresponding data and simulated curves for 11CB at smectic temperatures of 326.3 and 330.3 K, respectively.

In the absence of sound, if the linear dispersion model (N approach) is assumed to be correct, fittings using Eqs. (1) and (3) indicates that the self-diffusion mechanism completely determines the $T_1(\nu)$ behavior in all curves, i.e., the presence of the ODF relaxation mechanism cannot be detected from the measured dispersion curves. This may be the case if the mechanism is masked by self-diffusion. But if this argument was correct, there would not be any difference between dispersion curves in the presence and absence of ultrasound, unless the ultrasonic field was able to pull-up the ODF contribution at considerable strength. In addition, the observed behavior in the presence of sonication cannot be explained through fittings using the nematic approach [Eq. (13) for the ODF contribution]. On the contrary, if the smectic character of the mesophase is added, the behavior obtained is qualitatively well described, as can be seen in Figs. 1–4.

Simulated curves (except for 11CB at 330.3 K) were obtained through Eq. (1), where (14) was used for $T_{1OF}$. Due to the high quantity of involved parameters and the complexity of the function, typical values of constants $B$, $D$, $K_{11}$, $K_{22}$, $K_{33}$, $\eta$, and $a^2$ were used to generate simulated curves instead of data fitting (see Table I). Also, $\alpha$ and $\tau_D$ values were fixed (curves without sound were fitted without ODF contribution in order to obtain these approximated values). Other parameters were fitted, and they can be appreciated in Table II.

In the particular case of 11CB at 330.3 K, it was found that individual motions were governed by rotations. Then, the simulated curves were obtained following the equation:

$$\frac{1}{T_1} = b_1 + b_2 + C_D,$$

(17)

where

$$\frac{1}{T_{1Rot}} = A_R \left[ \frac{\tau_R}{1 + (2\pi \nu \tau_R)^2} + \frac{4\tau_R}{1 + (4\pi \nu \tau_R)^2} \right]^{-1},$$

(18)

being $A_R$ a constant, $\tau_R$ the correlation time for rotations, and $C_D$ a constant that represents the translational diffusion relaxation mechanism. This result is a consequence of the typical $\tau_D$ low values near the SmA–I phase transition ($\tau_D \sim 10^{-11}$ s, in most compounds). Then, $2\pi \nu \tau_D \ll 1$ in the whole Larmor frequency range of the measurements, giving a constant behavior for $T_{1TR}^{-1} = C_D$.

V. DISCUSSION

Considering we are comparing different compounds, it is important to note that $\tau_R$ and $\tau_D$ values obtained in the present work show a reasonable agreement with previous values obtained in the smectic phase of terephthalal-bis-p-butylaniline (TBBA). Another important fact is that the $\alpha$ values obtained in the smectic phase for both compounds exhibit the same order of magnitude as the one obtained by Vilfan et al. in TBBA ($\langle r^2 \rangle/\alpha^2 = 0.04$).

$T_1$ relaxation dispersion data are consistent with a model where individual motions are the dominant relaxation mechanisms. It was found that ODF relaxation dispersion relative weight for 8CB ranges from 3% to 9% at higher

| $T$ [K] | $A_D$ [$10^{15}$ s$^{-1}$] | $A_R$ [$10^6$ s$^{-1}$] | $\beta$ [$10^{14}$ s$^{2}$N$^{-2}$ m$^{-2}$] | $C_D$ [s$^{-1}$] | $\alpha$ [$10^{-2}$] | $\tau_D$ [$10^{-10}$ s] | $\tau_R$ [$10^{-8}$ s] |
|-------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| 295   | 4.3±0.9         | ...             | 1.4±0.2         | ...             | 1               | 2.4             | ...             |
| 298   | 5.5±0.7         | ...             | 1.6±0.8         | ...             | 1               | 2.0             | ...             |
| 301   | 7±1             | ...             | 1.5±0.2         | ...             | 1               | 1.6             | ...             |
| 304   | 6.5±1           | ...             | 1.2±0.2         | ...             | 1               | 1.6             | ...             |
| 326   | ...             | 5.0±0.3         | ...             | 6.2±0.4         | ...             | 1.6±0.2         | ...             |

| 8CB   | 326.5           | 6.5±0.6         | ...             | 17±3            | ...             | 2               | 2.5             |
|       | 330.3           | ...             | 2.2±0.3         | 2.0±0.4         | 5.8±0.6         | ...             | 2.1             |
|       | 340.0           | ...             | 2.8±0.2         | ...             | 6.2±0.4         | ...             | 0.95±0.09       |

11CB

| $T$ [K] | $A_D$ [$10^{15}$ s$^{-1}$] | $A_R$ [$10^6$ s$^{-1}$] | $\beta$ [$10^{14}$ s$^{2}$N$^{-2}$ m$^{-2}$] | $C_D$ [s$^{-1}$] | $\alpha$ [$10^{-2}$] | $\tau_D$ [$10^{-10}$ s] | $\tau_R$ [$10^{-8}$ s] |
|-------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| 326.5 | 6.5±0.6         | ...             | 17±3            | ...             | 2               | 2.5             | ...             |
| 330.3 | ...             | 2.2±0.3         | 2.0±0.4         | 5.8±0.6         | ...             | 2.1             | ...             |
| 340.0 | ...             | 2.8±0.2         | ...             | 6.2±0.4         | ...             | 0.95±0.09       | ...             |
frequencies and from 15% to 25% at lower frequencies. For 11CB these values are significantly lower at 326.5 K (1.8% and 8% at higher and lower frequencies, respectively). This result is consistent with the relative ratio \( n_c/n_t \) (where \( n_c \) is the number of protons in the core and \( n_t \) is the number of protons in the tail). It was also found that this relative weight does not present a strong dependence with temperature.

Even though a qualitative agreement is clearly observed between results and the proposed model, there is an effect observable in the experimental data and not explained by the model: The ODF contribution to the relaxation dispersion under sonication is shifted to lower relaxation time values compared to the experimental data at lower frequencies. For 8CB contribution from rotational diffusion only contributes in the SmA phase under sonication, as shown in Fig. 5. Relaxation dispersion and fitting curves for 8CB (a) and 11CB (b) in the absence of sound at the I phase temperatures of 326 and 340 K, respectively. It is possible to observe the contribution of rotational diffusion (RD) and translational diffusion (TD) mechanisms in both \( T_1(\nu) \) dispersions.

FIG. 5. Relaxation dispersion and fitting curves for 8CB (a) and 11CB (b) in the absence of sound at the I phase temperatures of 326 and 340 K, respectively. It is possible to observe the contribution of rotational diffusion (RD) and translational diffusion (TD) mechanisms in both \( T_1(\nu) \) dispersions.

VI. CONCLUSIONS

In this work we have shown that the \( T_1(\nu) \) dispersion in the SmA phase of cyanobiphenyls is consistent with a model where translational diffusion is the dominant mechanism. We also have shown that rotational diffusion starts to be relevant close to the SmA–I phase transition, possibly being a consequence of that.

The measurement of relaxation dispersion with the sample subjected to ultrasound allows detecting the presence of the ODF mechanism and quantifying its relative weight. Two previously proposed models were used to explain these data. It was found that a relaxation model including particular features of the smectic phase can be better used to describe the observed experimental data.

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