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Supra-Molecular Structure of TGBC* Phases Studied by Means of Deuterium NMR Line-Shape Analysis

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Supra-Molecular Structure of TGBC* Phases Studied by Means of Deuterium NMR Line-Shape Analysis

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In this work the structure of the tilted TGBC phase is investigated by means of the line-shape analysis of Deuterium NMR spectra. The case of a rod-like mesogen containing a (S)-2-methylbutyl-(S)-lactate unit in the chiral chain, whose acronym is* ***HZL 7****/*, *is taken as a representative example for the large temperature range of stability of its tilted TGB* phases. By comparing the experimental and computed Deuterium NMR spectra several structural features can be extracted. For instance, the analysis of Deuterium NMR line-shapes put in evidence the occurrence of helical deformations due to the effect of the external magnetic field.*

Keywords: banana-shaped; deuterium NMR; magnetic field; rod-like; smectic; twist grain boundary

INTRODUCTION

In Soft Matter there are several examples of frustrated phases [1] characterized by complex supra-molecular structures, isotropic ones, such as the blue liquid crystalline phases [2,3], and anisotropic ones, such as the so called twist grain boundaries (TGB) liquid crystalline phases [1,4,5]. Not tilted (TGBA*) and tilted phases (TGBC* and TGBC) have been discovered in different kinds of materials [1], but the supra-molecular structure of some of the tilted TGB phases has not been clarified yet [6]. From the theoretical point of view, several

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models have been proposed [4,7,8] and probably different structures really exist in different liquid crystals. For instance, two types of tilted TGB, one with a SmC*-type of structure within each block (TGBC*) [4], and another type without in-block helical structure (TGBC) [9] have been observed.

The characterization of the structure of both TGBC and TGBC* phases has been done by means of different experimental techniques, such as calorimetry [8], dielectric [10], optic [11] and X-ray measurements [12,13]. However, no NMR studies have been published so far. Few works are known about structural studies based on NMR spectroscopy of the TGBA* [14,15,16] and only a recent work has been done on TGBC* phases [17].

Another interesting issue related to TGB* phases is the response of their structure to external electric fields [18,19], which can totally unwind or strongly distort the helical supra-molecular structure. The effect of the magnetic fields have been investigated only recently on several liquid crystals, and in particular on the SmC* phase [20,21]. This mesophase structure can be completely or partially unwound by external magnetic fields higher than few Tesla [20,21]. The occurrence of the unwound SmC* (uSmC*) phase, as well as the appearance of the re-entrant smectic C phase, as a consequence of the presence of external magnetic fields, has been the object of several experimental and theoretical studies, showing that the value of the critical magnetic field H_C able to unwind the SmC* helices depends on the angle θ between the helical axis and the applied magnetic field, H [22]. The TGBC* phase, being constituted by blocks with SmC*-type structure, is expected to be very sensitive to the presence of external magnetic fields, as it is also shown in this work.

To this aim, Deuterium NMR spectroscopy represents a very useful tool giving important pieces of information about the mesophase structure and its stability in presence of the magnetic field. Deuterium NMR has been widely used to study common liquid crystalline phases [23], such as Nematic and Smectic A phases [24,25], but also more complex frustrated mesophases, such as the re-entrant smectic C* [26]. These studies confirmed the high potentiality of Deuterium NMR spectroscopy in order to clarify basic aspects about the structure and aggregation [27,28] of liquid crystalline phases.

In this paper, we show an application of Deuterium NMR spectroscopy to get information about the structure of the TGBC* phase, confirming the presence of the helical TGB twist and the SmC*-like structure inside each TGBC* block. Moreover, the simulation of the Deuterium NMR line-shapes further confirmed that the magnetic field is able to distort or unwind the in-block SmC* structure.

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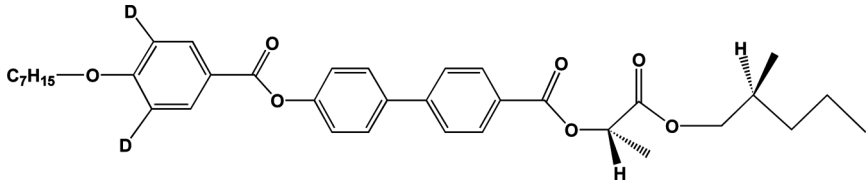
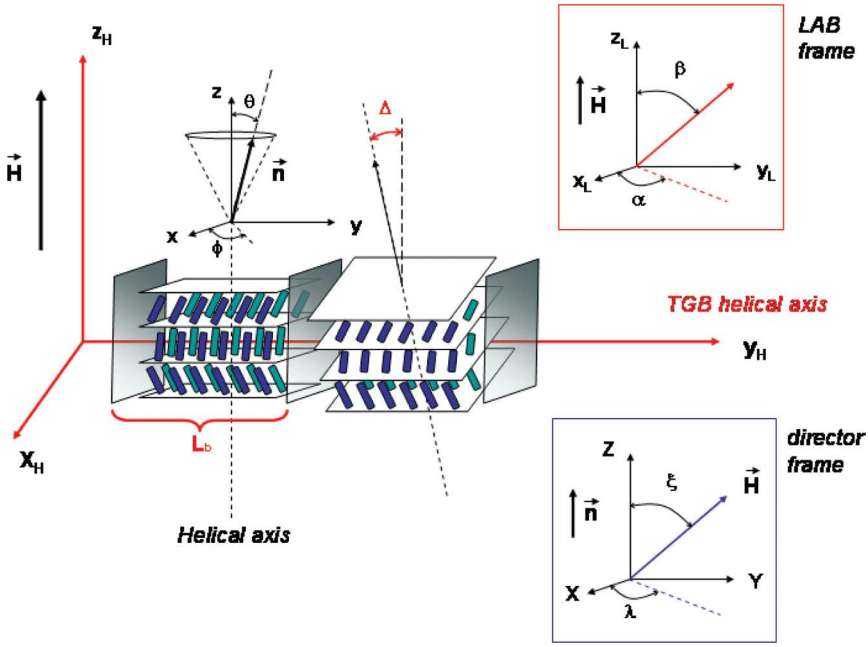
THEORY

The structure of the TGBC* phase proposed by Renn and Lubenski [4] and showed by the chiral compound **HZL 7**/*, whose structure is reported in Scheme 1, is characterized by a double twisted supramolecular structure. As it is shown in Scheme 2, the typical TGB structure is characterized by smectic blocks divided by grain boundaries which are uniformed distributed along one direction (y_H). The structure of each smectic block reminds that of the SmC* phase, with a helical axis (indicated with "z" in Scheme 2) always perpendicular to the TGB axis (y_H). As in common SmC* phases, the local director **n** describes an helical structure along "z", by varying the azimuthal angle $\phi(z)$ and maintaining constant the tilt angle θ. Contiguous TGB blocks are separated by grain boundaries, which represent the periodic element of the TGBC* phase with a constant distance equal to L_b. Moreover, two next TGB blocks are twisted of an angle equal to Δ which is typical of the specific compound and may also depend on the temperature. The angle Δ is between the two local SmC* helical axes of two adjacent TGB blocks (see Scheme 2).

For chiral molecules with a positive anisotropic magnetic susceptibility ($\Delta\chi = \chi_{\parallel} - \chi_{\perp}$), the orientation of the TGBC* domains with respect an external magnetic field is not random, but, according to the energetic contributions to the elastic energy of the system, it can be demonstrated [29] that the TGBC* axes (y_H) tend to align perpendicular to the magnetic field, H [30], similarly to the case of not tilted TGBA* phases [31] (see Scheme 3). The resulting distribution of domains is cylindrical with respect to the direction of the magnetic field, H (z_H in Scheme 2), which is parallel to the z_L axis of the laboratory frame. The helical pitch, namely the TGB axis, forms always an angle $\beta = \pi/2$ while the angle α ranges between 0 and 2π. However, if the magnetic field is strong enough [17], either the helical TGB structure or the in-block SmC* structure, in the case of TGBC* phase, can be distorted or completely unwound.



SCHEME 1 Molecular structure of the rod-like mesogen **HZL 7**/* showing several TGB* phases. This sample was selectively deuterated on the phenyl ring.



SCHEME 2 Scheme of the supra-molecular structure of the TGBC* phase. Each block is characterized by SmC* phase structure (x, y, z frame), being the SmC* helical axis indicated by "z". The TGB frame is x_H, y_H and z_H, and the TGB helical axis is along "y_H". The NMR Lab frame is indicated as x_L, y_L and z_L, and the external magnetic field H is along "z_L". α and β refer to the relative polar angles defining the TGB helical axis respect to the LAB frame; ϕ and θ refer to the relative polar angles defining the director **n** respect to the in block SmC* frame. L_b and Δ are the dimension of the TGB block and the jump between two next blocks, respectively. The director frame is also shown (X, Y, Z).

Deuterium NMR spectra are very sensitive to the orientation of the molecules with respect to the magnetic field, thus the occurrence of peculiar distributions, such as in the case of TGB structure strongly affects the resulting NMR spectra. Among different contributions, the quadrupolar interaction is the most important one in Deuterium NMR of anisotropic systems and the Hamiltonian can be written as [23]:

$$H_Q = \frac{e^2qQ}{4I(2I-1)}[3I_Z^2 - I(I+1) + \eta(I_+^2 + I_-^2)/2] \quad (1)$$

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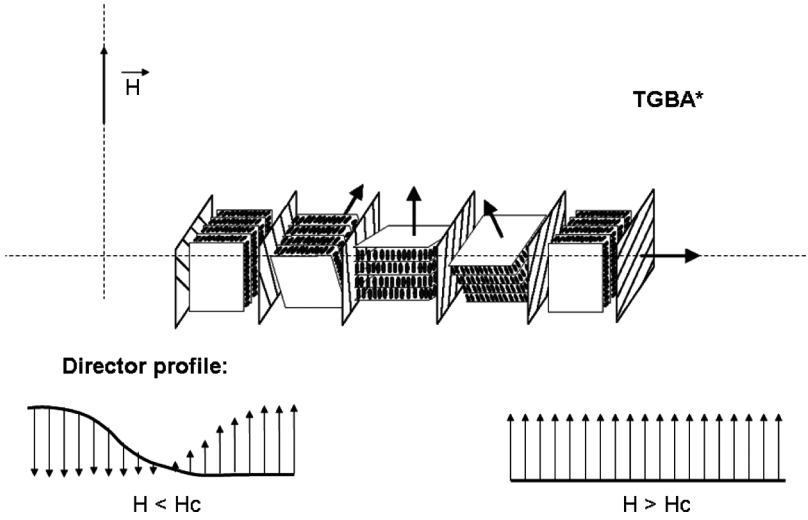

SCHEME 3 A schematic picture of the TGBA* phase. The alignment of the helical axis in conventional liquid crystal is perpendicular to the magnetic field, H. Depending on the relative value of the field, H, respect to the critical field, H_C, the helical supra-molecular structure can be slightly deformed (on the left) or completely unwound (on the right).

where *eq* is the principal element of the electric field tensor $\hat{V}$ along the direction *Z*, and the term η is equal to $(V_{XX} - V_{YY})/V_{ZZ}$. In liquid crystalline systems, molecules are affected by fast reorientational and translational motions and the averaged quadrupolar Hamiltonian yields to a spectrum dominated by a doublet with frequencies:

$$\nu_{\pm} = \frac{3}{4}\bar{\nu}_Q\left(\frac{3}{2}\cos^2(\xi) - \frac{1}{2} + \frac{\bar{\eta}}{2}\sin^2(\xi)\cos(2\lambda)\right) \tag{2}$$

where $\bar{\nu}_Q$ and $\bar{\eta}$ are the time averaged coupling constant and asymmetry parameter, respectively, defined in the local director frame, and the angles ξ and λ are the polar angles describing the orientation of the magnetic field, H, in the local director frame.

The Deuterium line-shape can be simulated numerically by calculating the Fourier transform [23]:

$$I = I_0 \int_0^{\infty} G^{\pm}(t) \cdot \cos(2\pi\nu^{\pm}t) \cdot dt \tag{3}$$

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where $G(t)$ is the free induction decay which can be expressed as:

$$G^{\pm}(t) = G_0 \int_0^{2\pi} \int_0^{2\pi} \sum_0^s W(\theta, \phi, n\Delta, \alpha) \cos[2\pi\nu^{\pm}(\theta, \phi, n\Delta, \alpha)t] \cdot R(\sigma,\ t) \cdot d\phi \cdot d\alpha \tag{4}$$

with

$$R(\sigma, t) = \exp(-\sigma^2 t^2/2) \tag{5}$$

In Eqs. (4) and (5), R is the function introducing the Gaussian line-broadening with relevant parameter σ, $W(\theta, \phi, n\Delta, \alpha)$ is the weight for the specific orientation at the constant tilt angle θ, and variable angles α, ϕ and $n\Delta$ (with $s = 2\pi/\Delta$). The frequency $\nu^{\pm}(\theta, \phi, n\Delta, \alpha)$ is a function of the constant tilt angle θ, and variable angles α, ϕ and $n\Delta$ with the following coordinate transformations from the local director frame (X, Y, Z, with **n** along the Z axis) and the Laboratory frame (x_L, y_L, z_L) (see Scheme 2):

Local director frame (X, Y, Z) $\rightarrow (\theta, \phi) \rightarrow$ SmC$*$ frame (x, y, z)

SmC$*$ frame (x, y, z) $\rightarrow (0, n\Delta) \rightarrow$ TGBC$*$ frame (x_H, y_H, z_H)

TGBC$*$ frame $(x_H, y_H, z_H) \rightarrow (\alpha, \pi/2) \rightarrow$ LAB frame (x_L, y_L, z_L)

—

Local director frame (X, Y, Z) $\rightarrow (\xi, \lambda) \rightarrow$ LAB frame (x_L, y_L, z_L)

In the case of undistorted structure the weight function $W(\theta, \phi, n\Delta, \alpha)$ is equal to 1 for any groups of angles defining the orientation of the local director with respect to the magnetic field. However, it is well known that the magnetic field can induce a deformation of the director phase profile. A typical example is that of a simple SmC* phase, where the only variable angle describing the director orientation is the azimuthal one, $\phi(z)$: The fraction of the helix for which the angle between the molecular director and the magnetic field is the smallest increases. For example, considering the function $\phi(z)$ in the case of a smectic helix perpendicular to the magnetic field, with $\theta = 90^\circ$, which would be equivalent to a cholesteric phase, the deformation provides a 2π-soliton lattice. A possible analytical expression describing the function $\phi(z)$ is given by the following equation [29,32]:

$$\phi(z) = z + a[\sin(2z) + 1/4 \cdot \sin(4z) + 1/45 \cdot \sin(6z)] \tag{6}$$

where a is an empirical parameter increasing with increasing distortion, ranging from 0 to 3/4 [32]. The effect of the distortion of the

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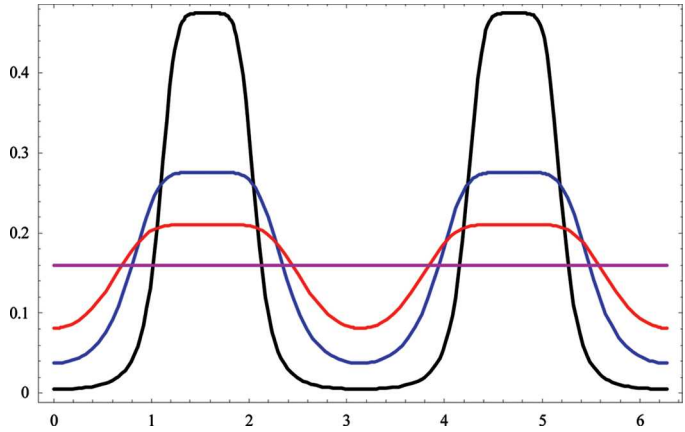

FIGURE 1 Weight-function of the different orientation (azimuthal angle ϕ from 0 to 2π) of the local director **n** depending on the deformation of the in-block SmC* phase structure. Pink lines refer to zero deformation (all orientation are equally populated) ($a = 0$); red, blue and black functions refer to deformed distributions with $a = 0.25$, $a = 0.5$ and $a = 0.75$, respectively.

phase profile can be taken into account starting from equation (6) and computing different weights $(d\phi(z)/dz)$ for different orientations of the local director with respect to the magnetic field. Some examples of weight functions for different values of the parameter a are reported in Figure 1. By introducing this kind of deformation is possible to compute the spectral Deuterium NMR line-shape and the effect is evident as reported in the following section.

RESULTS AND DISCUSSION

Some examples of Deuterium NMR spectra of the mesogen **HZL 7**/* in its TGBC* phase are reported in Figure 2. At higher temperatures, the sample **HZL 7**/* shows a TGBA* phase and the effect of the cyndrical distribution of orientations determines a typical spectral shape, which is indeed quite similar to the cholesteric one [16]. If the external magnetic field, H, is higher than the critical field, Hc, the helical structure of the TGBA* phase is unwound (see Scheme 3) and the local directors are oriented on average parallel to the external magnetic field. In the latter case, the NMR spectrum is dominated by a single doublet [17].

The case of the TGBC* phase is much more complicated, because two twisted structures are present at the same time. If the magnetic

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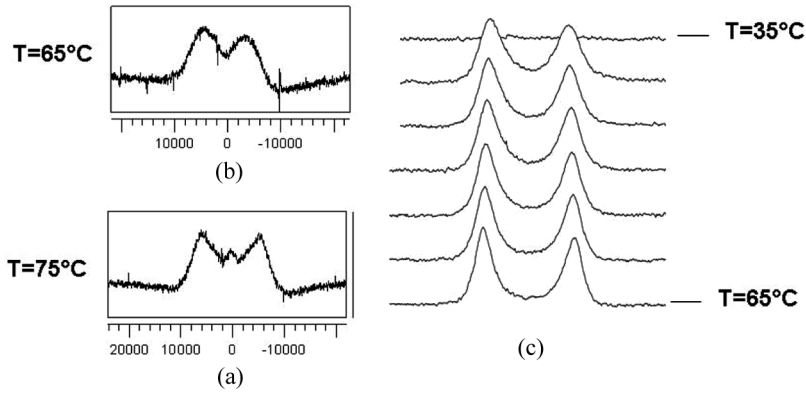

FIGURE 2 (a) and (b) Experimental Deuterium NMR spectra of the mesogen **HZL 7**/* in the TGBC* phase, recorded at 4.70 Tesla ($H<H_C$). (c) Experimental Deuterium NMR spectra of the mesogen **HZL 7**/* in the TGBC* phase, recorded at 9.40 Tesla ($H>H_C$).

field H is strong enough to unwind both helical structures, the spectrum is very simple, because it is characterized by a single doublet, as reported in Figure 2c. Different is the case of partial deformations or unwinding of only one the two supra-molecular helical structures. In order to better clarify the orientational behaviour of the TGBC* phase in the presence of the magnetic field, we can compute the Deuterium NMR line-shape corresponding to different assumptions, first without introducing any distortions and then by including the distortion or/and unwinding of the in-block SmC* structure.

The knowledge of the experimental value of the tilt angle, θ, for the sample **HZL 7**/* at the temperatures [15] of the recorded spectra (Figs. 2a and 2b) is of help when using Eqs. (3)–(6) in order to compute the Deuterium NMR line-shape. In fact, the value of the tilt can be fixed to 35° [5]. Assuming a step Δ of 3 degrees, thus with very small jumps among near TGB blocks, without any distortion of the in-block structure, the resulting spectrum is that reported in Figure 3a. As demonstrated in a previous work [32], the tilt angle θ of the SmC* phase heavily influences the expected line-shape. In fact, the "Pake-like spectra" for an isotropically distributed SmC* phase with a tilt angle different from 0 are very different from the "Pake spectrum" due to an isotropic distribution of molecular directors. Taking as a constant the value of the tilt angle, according to the experimental results [15], we introduced the effect of partial

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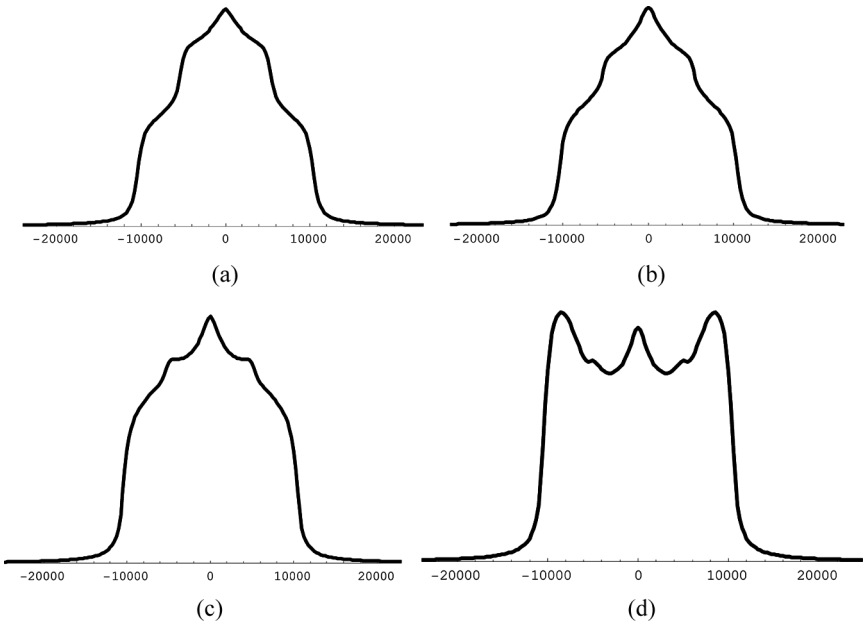

FIGURE 3 Simulated Deuterium NMR spectra assuming a phase order parameter S of 0.6, a tilt θ of 35 degrees, small jumps between a block and another ($\Delta = 3$ degrees), Gaussian shape, and a gradually increased level of deformation of the in-block helical distribution: (a) $a = 0$; (b) $a = 0.25$; (c) $a = 0.5$ and (d) $a = 0.75$.

unwinding of the SmC* helix, by varying the parameter a. A series of computed spectra are reported in Figures 3b–3d. By comparing these computed spectra with the experimental spectrum of Figure 2a, we can notice that the appearance of lateral external peaks may be correlated to the higher deformation of the director distribution, while the presence of the internal central peak is due to the particular value of the tilt angle [32].

Another interesting effect is that related to the number of TGB blocks per pitch (n), or the angular jump between close TGB blocks (Δ). In fact, in the literature several cases of different structures for the TGBC* phase are reported [1,7,8,13], including the commensurate and incommensurate ones, whose difference is related to Δ [6]. The effect of the combined effect due to the number of blocks and the deformation of the SmC* helix by the external field is shown in Figure 4. Here, it is evident that the decreasing of n determines the disappearance of the singularities: the signals result

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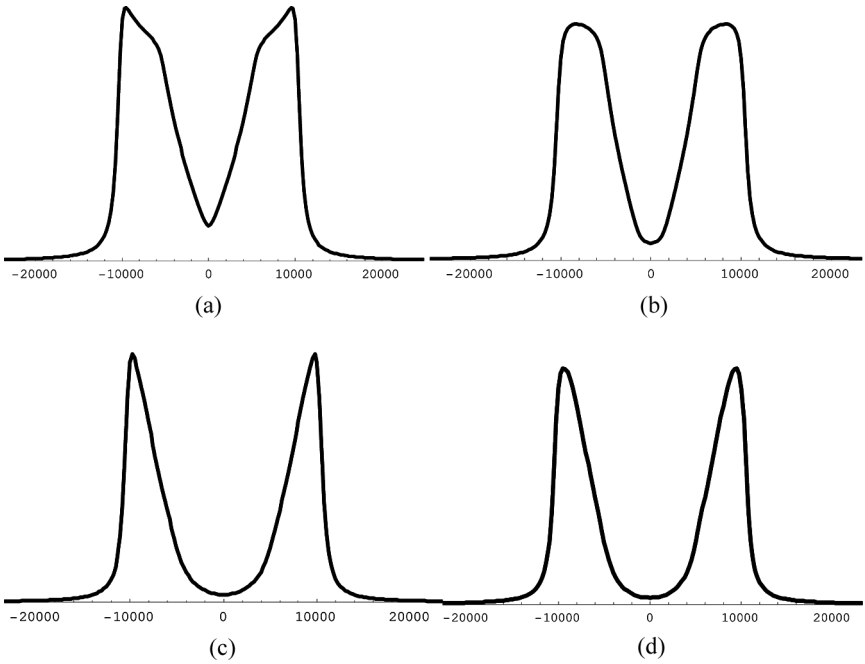

FIGURE 4 Simulated Deuterium NMR spectra showing the effect of the number of TGB blocks per pitch and the effect of the in-block SmC* deformation. (a) $a = 0$ and 30 blocks; (b) $a = 0$ and 20 blocks; (c) $a = 0.75$ and 30 blocks; (d) $a = 0.75$ and 20 blocks.

to be less structured and smoother. If we compare these computed spectra with the experimental spectrum reported in Figure 2b, we can observe a similarity between this spectrum and that in Figure 4b. A qualitative final observation concerns the claiming of the occurrence [15,17] of two different TGBC* phases passing from $T = 75°C$ and $T = 65°C$, corresponding to the experimental spectra reported in Figures 2a and 2b. According to the change in the NMR line-shape the first phase ($TGBC_1^*$), at $T = 75°C$, is characterized by a strong deformation of the in-block SmC* phase, with very small steps among close TGB blocks (almost incommensurate) [6]. On the contrary, the second phase ($TGBC_2^*$), at $T = 65°C$, seems to be characterized by any deformation of the in-block SmC* phase, with a number of steps, n, much more typical of TGB* structures [6].

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CONCLUSIONS

In this work, the supra-molecular structure of the frustrated liquid crystalline TGBC* phase, under the effect of external magnetic fields, have been studied based on Deuterium NMR spectral analysis. The analysis of the Deuterium NMR line-shape is a useful tool in order to verify the structural models proposed for the tilted TGB* phases. In the examined case, the effect of the magnetic field is quite strong mainly on the in-block helical structure. In fact, this study revealed that the typical TGB structure, characterized by the helical distribution of TGB blocks, is retained, while the in-block SmC* phase structure may be strongly distorted. The number of blocks per TGB pitch can also affect the Deuterium NMR line-shape. We propose a possible explanation about the structural differences among two types of TGBC* phase shown by the liquid crystal **HZL 7**/*.

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