The Physics of Lyotropic Nematic Liquid Crystals

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Lyotropic nematic liquid crystals are rnixtures of amphiphilic molecules and a solvent which under particular temperature- concentration conditions present some degree of orientational ordering of the micelles. In this paper we present an overview of the research in this area, focusing on **problems** like structure, local ordering, phase transitions, anchoring and hydrodynarnic instabilities in lyotropics. The change in the shape anisotropy of the micelles as a function of temperature is discussed as being responsible for many unusual behaviors observed in experiments with lyotropics.

I. Introduction

The intermediate phases between the solid crystal and the isotropic liquid are defined by means of their syrnmetry properties and degree of order.

Plastic crystals' are mesophases whose building-blocks (atoms or rnolecules) present tridimensional order of the centers of mass (characteristic of the crystalline lattice) and, also, orientational disorder.

Liquid crystals are mesophases whose building-blocks present many different degrees of orientational ordering and, in some examples, even positional ordering[2]. Tlie majority of the substances which present liquid crystal phases have elongated molecules[2]. Chandrasekhar and co-workers[3] showed that disk-like molecules present also liquid crystal phases.

Liquid crystals are divided in two main categories: the thermotropics and the lyotropics.

Thermotropic liquid crystals are usually obtained by heating a solid substance composed by anisometric molecules (elongated or disk-like). They present an isotropic phase at high temperature. The relevant parameters for the phase transitions are temperature and pressure[4]. These siibstances are used in many liquid crystal devices as thermometers, displays etc.' .

Under proper temperature-concentration conditions, mixtures of amphiphilic molecules and a solvent (usually water) give liquid crystalline mesophases called lyotropics[2]. The temperature, pressure[5] and the relative concentrations[4] are the relevant parameters which are responsible for the phase transitions. The amphiphilic molecules are composed by a polar head and a paraffinic chain. When these molecules are put in contact with some solvent (polar or not), at a concentration higher than the critical micellar concentrations (cmc), aggregates of molecules are formed. These aggregates can have different forms and when their shape anisotropy (anisometry) is small, they are called micelles. The micelles can be considered the building-blocks of the lyotropic liquid crystals.

Structural investigations of liquid crystalline mesophases are done essentially by means of optical and scattering techniques[6,7,8,9,10].

The phase diagrams of liquid crystals are usually very complex[4,11]. Many different phases exist in liquid crystals as a function of temperature (and other convenient parameters), with typical structures and symmetry. One of these phases is the NEMATIC one. This name, from Greek origin, was proposed by Friedel[12] in 1922 and is related to the line-shaped defects which were observed in its texture, under a polarizing microscope. In the nematic order, the centers of mass of the building-blocks of the liquid crystals do not present long range positional ordering. However, they present a long range orientational ordering. The positional correlation among centers of mass of molecules (or rnicelles) is similar to the one of isotropic fluids.

Liquid crystals in the nematic phase are largely used in displays[1].

Another kind of molecular ordering which was found in liquid crystals is the CHOLESTERIC one. In this phase, the molecules (or micelles) twist[2] in a scale of the order of 1μm around a particular direction. On this condition, the helical structure is achieved.

Regarding symmetry properties, the nematic phase can be considered a cholesteric one of infinit helical pitch.

As well as these kinds of molecular ordering, liquid crystals also present many others[1,2] such as the smectic (lamellar), the hexagonal and the cubic order.

In this paper we will focus our attention on the lyotropic nematic ordering. It is summarized the most important contributions of the São Paulo group in the field.

II. Microscopical structure of lyotropic nematics

From symmetry reasons[13], three kinds of nematic phases are expected to exist: two unaxial and one biaxial. In 1980, Saupe and co-workers[14] investigated the phase diagram in the lyotropic mixture of potassium laurate, decanol, water and observed the three nematic phases for the first time. The uniaxial phases are called[9] calamitic (N_C) and discotic (N_D) and the biaxial phase N_B. From x-ray[9] and neutron[15] diffraction measurements, the N_C and N_D phases were first concluded to be, respectivelly, made of prolate and oblate micellar aggregates dispersed in water. Within this scheme, several experimentalists have believed that the transition from the uniaxial to the biaxial nematic phase was produced by a simple change in the micellar shape. From the uniaxial N_D (or N_C) to the biaxial nematic phase transition, the micellar shape was supposed to change from a flat disk (or an elongated cylinder) to a biaxial flattened ellipsoid. The picture of disks and rods was proposed for the first time by Reeves and co-workers[16] on the basis of nuclear magnetic resonance (NMR) measurements and considerations of the anisotropy of diamagnetic susceptibility of the phases. The first x-ray diffraction patterns[9] obtained with the uniaxial phases were then interpreted assuming a static configuration, neglecting the possible orientational fluctuations of the micelles. At that time, the N_B phase had not been observed yet. In 1983, Galerne and Marcerou[17] studied the uniaxial to the biaxial phase transition measuring the optical birefringence. Their results indicate that these transitions are second-order, with a correlation length ξ (concerning the biaxial order) always larger than the micellar size. The theory of second-order phase transitions shows that the local ordering, on scales smaller than ξ, should be the same on both sides of the transition, and identical to the one of the ordered phase. Independent Rayleigh's experiments[18] estimate the bare correlation length ξ_0 at the $N_D - N_B$ transition is about 100 Å. This indicates that

$$\xi = \xi_0 \left(\frac{T - T_c}{T_c} \right)^{-1/2} \qquad (1)$$

is larger than ~ 500 Å over the whole nematic range, at least an order of magnitude larger than the typical micellar size[15] (about 50 Å). So, in this picture, drastic changes on the micellar shape (from disks to rods) are not expected in the phase transitions.

At that time, new x-ray diffraction experiments were required to clarify the structure and the local ordering of the nematics.

The lyotropic investigated[10] was a mixture of potassium laurate, decanol and water, prepared according conventional procedures[11]. The composition in weight percent is: 26.50/6.68/66.82 respectively. As the temperature increases, the successive phases identified by conoscopic measurements[17] were observed: isotropic ($9.5°C$), $N_D(13.2°C)$, $N_B(23.2°C)$, $N_C(40°C)$, isotropic. The sample was sealed in Lindemann glass capilaries, placed in a temperature controlled device (accuracy of $0.1°C$). A magnetic field was applied perpendicular or parallel to the x-ray beam, depending on the particular plane of the reciprocal space investigated. X-ray diffraction patterns were obtained using the synchrotron x-ray monochromatic radiation (Ge crystal, wavelength $\lambda = 1.62$ Å) of Orsay (Laboratoire pour l' Utilisation du Rayonnement Electromagnetique - LURE). The geometry of the experiment is the classical Laue. The experimental resolution of the setup $\Delta q(= 2\pi \Delta s$ where $s = [2 \sin \theta]/\lambda$ and 2θ is the scattering angle) were : 9×10^{-4} and 1.5×10^{-3} Å^{-1} along the vertical and horizontal directions respectively.
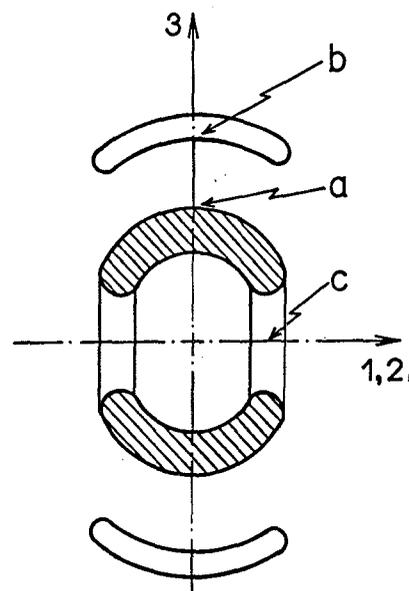
Figure 1: Sketch of the X-ray diffraction patterns in the oriented nematic phases.

The X-ray patterns obtained in monodomains of the N_D, N_C and N_B phases were remarkably similar: (see for example figure 1 on ref. 10 and figures 3, 4 on ref. 19). The diffraction patterns are essentially composed by three bands. Figure 1 sketches the X-ray diffraction patterns in the oriented nematic phases. The (a) and (b) bands are due to the pseudo-lamellar ordering and the (c) band is related to micellar positional ordering along the direction perpendicular to the amphiphilic bilayer. The reciprocal structure of the uniaxial phases may be schematized[10] as a hollow cylinder parallel to the direction of the infinite-fold symmetry axis (optical axis) of the phase, with intense ends in the N_D phase, and intense edges in the N_C phase. The biaxial phase has three perpendicular two-fold axis, and the reciprocal structure of the N_B phase is a hollow barrel of a flattened section. In all patterns of all nematic

phases, it was observed that a second-order band appears with a spacing ratio of 1/2 associated to the first order band due to the amphiphilic bilayer. This fact indicates that the three nematic phases show the same pseudo-lamellar structure on short range scales.

Table I

Summary of the typical parameters of the potassium laurate/decanol/water lyotropic liquid crystal.

Micellar dimensions (Å)	A'=85	B'=55	C'=26
Dimensions of volume available per micelle (Å)	A=110	B=80	C=49
micellar shape anisotropy	3	2	1
characteristic length(Å)	$\xi_A = 70$	$\xi_B = 130$	$\xi_C = 300$

The careful analysis of the diffraction bands (ever their structure) indicated that the micelles have a biaxial symmetry[10,19,20,21] in the three nematic phases. The different nematic phases are then the macroscopical consequence of different orientational fluctuations of the micellar correlation volumes[2]. These orientational fluctuations are full rotations around the infinite-fold symmetry axis in the uniaxial phases and small amplitude oscillations in the biaxial nematic phase. In this picture, the micelles in all the lyotropic mixtures of two amphiphiles and a solvent have, on average, the shape of biaxial platelets[21]. In particular[20], in the potassium laurate/decanol/water mesophase, these biaxial platelets have a shape anisotropy of about 1/2/3. The typical dimensions of the micelles (potassium laurate mixture) are about $A' \simeq 85$ Å, $B' \simeq 55$ Aand $C' \simeq 26$ Å in the middle of the N_B temperature range. C' in this case is the potassium laurate amphiphilic bilayer. The characteristic length obtained from the width at half height of the diffraction bands (positional correlation length=ξ) varies depending on the considered direction: in the direction perpendicular to the amphiphilic bilayer, $\xi_C \sim 300$ Å; in the plane of the amphiphilic bilayer, $\xi_A \sim 70$ Å and $\xi_B \sim 130$ Å. A possible source of anisotropy in these parameters is the distribution of decanol and potassium laurate on micellar surface. Neutron scattering studies[22] show that alcohol concentrates at the flat core of the micelle rather

than at the high-curvature rim. In the intrinsic biaxial micelles model we also expected different curvatures of the micellar borders in the plane of the amphiphilic bilayer. This anisotropic distribution of alcohol could be the responsible for modifications in the form factor of the micelles. Thus, the X-ray interference function could be modified by this effect.

Table I presents a summary of the typical parameters of ternary lyomesophase of potassium laurate.

In conclusion, the model of intrinsic biaxial micelles in all the uniaxial and biaxial nematic phases is consistent with all the experimental results extracted from the X-ray diffraction patterns, optical techniques, NHR, neutron scattering[23] and measurements of coefficient of thermal expansion on lyotropics[24].

III. Anchoring properties of lyotropic nematics

The anchoring properties of thermotropic nematics have been widely investigated in both experimental and theoretical points of view[25,26,27,28].

The knowledge of these properties, besides the scientific interest, is very important for technological applications of liquid crystals in displays[29,30].

Many models have been proposed[31] in the last years in order to understand these interface phenomena. In these models, it is usually considered an anisotropic elastic energy characterized by a parameter called *extrapolation length*. The extrapolation length (L) is inversely proportional to the intensity of the anchoring. In case of a weak anchoring, perturbations of the molecular orientation at the interface can occur under external fields. However, when the field is turned off, the orientation in the interface (and even in the bulk in thin samples) is dominated by the surface of the sample holder.

On the other hand, the anchoring properties of lyotropics have not been completely investigated yet. The study of the Frederikcs transition[32] in the mixture of potassium laurate indicates an unusual behavior of the orienting and the relaxation times under the influence of magnetic fields. Assuming the classical models of anchoring applied to thermotropics and the viscosity of lyotropics, the rising and the obtained relaxation times are orders of magnitudes, different from the experimental values[33].

The lyotropic samples used to investigate the anchoring properties were mixtures of potassium laurate/decanol/water and potassium laurate/decylammonium chloride/water. This second mixture[11] has the advantage of being very stable from the chemical point of view. The phase under investigation was the nematic N_C. .The sample holder was made of microslides 200μm of thickness and 4mm of width. The inner surfaces of the microslides were examined in the interferential microscope. No irregularities or channels greater than 0.1μm were observed. The

laboratory-frame axes were defined with the x axis parallel to the length of the microslides, and the z axis normal to the biggest surfaces. Initially, the sample is uniformly oriented[33] by a magnetic field (H) of about 18kG, parallel to the x axis. Then a magnetic field of different strength is applied at 45° from the x axis (perpendicular to z). The evolution of orientation of the sample was studied by measuring the extinction angle in a polarizing microscope, as a function of time. The same measurements were made during the relaxation process when H was switched off. In N_C samples, as the anisotropy of diamagnetic susceptibility (χ_a) is positive, the symmetry axis (usually called[2] the director $\vec{n}$) orients parallel to $\vec{H}$. Two different characteristic times were observed[33]: a short response time (about 20 s for H = 2kG), and a long response time of hours.

The orienting times r in the presence of H are shown in Table II. These values follow a $1/H^2$ law. They cannot be interpreted on the basis of the usual models known as thermotropics: the strong anchoring model predicts an orienting time of about 50 s but the times measured are about 900 s (H = 4.5 kG).

Table II

Orienting times (r) as a function of H.

H (kG)	$\tau(hours)$
3.0	0.57
3.8	0.34
4.5	0.25
6.0	0.12
8.0	0.07

The relaxation process also cannot be understood on the basis of the usual anchoring models. After the relaxation process, the equilibrium is reached to a final orientation at an angle different from zero, that depends on the time the magnetic field is applied. This means that $\vec{n}$ at the surface, rotates towards the direction of $\vec{H}$, and it does not return to its original orientation (parallel to x) when the magnetic field is switched off. This is a new phenomenon, not yet observed in thermotropics: the director glides at the surface, with a characteristic time that is also proportional to $1/H^2$ (Table III). These results are incompatible with the usual anchoring models that always gives[33] a characteristic time proportional to $1/H$.

Hence, an alternative model is required. Let us make the hypothesis that[33] at the surface, there is a bilayer of amphiphilic molecules, with defects or channels, like in a lamellar structure[34]. Initially, in the presence

of H, the micelles in the bulk are uniformly oriented at 45° and the alignement is perturbed in a layer of thickness ξ near the surface. The defects at the surface layer can be interpreted as large anisotropic objects that can glide and align parallel to $\vec{H}$ if they have surface dimensions of order ξ. If they are smaller, the competition between the elastic torque coming from the volume does not favor this gliding. Being these objects larger (their dimensions in the x-y plane are a few μm) than the micelles (dimensions around 10^2 Å), there is an apparent larger viscosity near the surfaces. These large objects are submitted to the elastic torque coming from the micelles in the bulk, which is equilibrated by the viscous torque.

Table III

Characteristic time of the gliding process τ_g as a function of H.

H (kG)	τ_g (hours)
4.5	16.9
8.0	6.5
15.0	1.2

The balance of torques at the surface gives[33]

$$\gamma \overset{\circ}{\theta_s} \alpha\xi = \frac{K}{\xi}\left[\frac{\pi}{4} - \theta_s\right], \qquad (2)$$

where γ is the viscosity, θ_s the angle between $\vec{n}$ and the x axis at the surface, a is the number of objects (channels of defects) per unit area and K is the elastic constant[1]. The solution of this equation gives an exponential increasing of θ_s with a characteristic time:

$$\tau_g = \frac{\xi^2 \gamma \alpha}{K}, \qquad (3)$$

and thus

$$\xi = \left(\frac{K}{\chi_a}\right)^{1/2} \frac{1}{H}, \qquad (4)$$

the $1/H^2$ law is obtained. The product $\alpha\gamma$ can be interpreted as an apparent viscosity.

According to the experimental point of view, some kinds of defects (such as grains) were observed in the textures[33]. It was also noticed that the size of these grains depends on the magnetic field. The typical dimensions of defects are about 30 x $80\mu m^2$ for H = 4.5kG. Small dimensions of defects were observed with a strong H, that confirms the hypothesis that the dimensions of the grains are about ξ.

With this qualitative model[33] the long period observed can be understood and also gives the correct

dependence of τ_g on $1/H^2$. The gliding of the director at the surface is an interesting phenomenon that is probably related to the particular characteristic of the lyotropic systems. It has never been observed in thermotropics. In lyotropics, the amphiphilic molecules at the surface probably cannot move, but in the bilayer structure, the defects on channels can be destroyed and rebuilt with a new orientation, due to the finite lifetime of the micelles.

Another experimental geometry was recently investigated[35]. In a discotic N_D lyotropic, $\vec{n}$ orients perpendicular to the glass surfaces. Then, H is applied to modify the orientation of $\vec{n}$. The experimental results obtained indicate that in this configuration there is a strong anchoring of the objects.

IV. Thermal instability in lyotropic nematics

The study of thermal instabilities in thermotropics has been made[36] from both experimental and theoretical points of view for several years. The main difference between this instability and the classical Rayleigh-Bénard of isotropic fluids is the existence of anisotropic parameters in liquid crystals. This instability is due to the thermal coiductivity anisotropy. So, the heat flux is not perpendicular to the samples' plates, but deviates along the molecules. Under the influence of the buoyancy forces, warmer and cooler regions move up and down, and the total viscous torque destabilizes the structure. The convection appears above a threshold whose value and sign depend on the initial configuration[37]. For a planar configuration, it is induced by a heating of the sample from below, whereas for a homeotropic one, the sample is heated from above. As in the isotropic case, the wavelength (A) of the roll pattern is about twice[36] the sample thickness (d), i.e., $qd = (2\pi/\lambda)d \simeq \pi$, considering the destabilizing effects of the surface-tension traction leads to the Bénard-Marangoni instability[38]. In these cases, always $A \approx d$.

Charvolin and co-workers[39] reported an experimental study of instabilities in a lyotropic nematic phase induced by a magnetic field (at a constant temperature). They obtained $10 \leq qd \leq 40$ and the observed roll structure occurred due to the back-flow effect induced by fluctuations of micellar orientation in the presence of H. Lonberg and Meyer[40] showed a new form of Fredericksz transition with a complex periodic twist-splay pattern, induced in a polymer-liquid-crystal sample. Also in these cases, $\lambda \approx d$.

To our knowledge, the first experimental study of thermal instabilities in lyotropic was reported in 1990. The lyotropic liquid crystal which was studied is the potassium laurate/decanol/water mixture, which has a N_C phase from 15°C to 50°C. This N_C phase is surrounded by two isotropic phases. Reentrant phases are usually found in phase diagrams of lyotropics[11,14].

We will come back to this point later on. The sample is encapsulated in glass cells with dimensions A = 4mm (parallel to the y axis), B = 2cm (parallel to x) and different thickness d (around 1mm) (parallel to z). Initially the sample is oriented with $\vec{n}$ parallel to x by applying a magnetic field (13kG). When a good planar alignment is achieved, the field is switched off and a thermal gradient is applied along z. The cold plate is maintained at a fixed temperature (T_{co}) and the hot plate temperature (T_h) can vary. AT is defined as the temperature difference $T_h - T_{co}$ along the z axis of the sample. The texture is observed in a polarizing microscope. Initially $T_{co} = T_h = 25°C$ and a planar texture was observed. After, T_{co} is maintained constant and T_h increases up to 44°C. If AT is kept constant, the sample remains planar. When $44 < T_h < 50°C$ starts to decrease, with T_{co} fixed, a periodic roll structure with stripes perpendicular to the x axis is observed when T_h reaches 44°C. If $T_h < 44°C$ ($15 < T_{co} < 44°C$) and then starts to decrease with $0.16 \leq \gamma (= |d(\Delta T)/dt|) \leq 0.72°C/min$, the instability is not observed. The same features are observed if T_h is at $z = 0$ and T_{co} at $z = d$ or if the sign of AT is changed. The periodic structure always begins to appear in the X-Y plane near the hotter plate. In a few seconds it extends along z. In case of a horizontal heating, the planar alignment gets lost, probably due to convective motion of only one big convective cell. The wavelength (A) of the periodic structure depends on the sample thickness and y [41]. For a fixed y, $q(= 2\pi/\lambda)$ increases for increasing values of d. For a fixed value of d = 1mm, q presented a linear behavior with y and a critical value of $\gamma_c = 0.1$ °C/min was obtained for $q^2 = 0$. Concerning the phase diagram in the plane (AT,y) (see Figure 4 of ref. 41), a minimum critical value of AT was determined: $\Delta T_c = 2°C$. From these values, a time constant $r_c = \Delta T_c/\gamma_c$ can be calculated: $\tau_c \sim 20$ min. It is related to the physical process that generates the instability. In these experiments[41] we have $30 < qd \leq 300$.

All the features which characterize this instability clearly indicates that it is different from the classical Rayleigh-Bénard convection.

When AT suddenly vanishes or is kept constant, the roll structure relaxes to the initial planar geometry. The relaxation time τ for y = 0.6°C/min, d = 1mm is about 8 min.

The optical microscopic investigation of the roll's texture showed that there is a bend distortion of the director in the X-Y plane. The observation of the movements of small grains of polystyrene (typical dimensions about 5μm) dispersed in the sample indicates two kinds of movements: up and down along the z axis and a back-and-forth movement perpendicular to the stripes. These movements are consistent with a family of rolls with the axes parallel to the stripes[41] (Figure 2). In these rolls, the micelles seem to be in a plane tilted from the z axis. The tilt angle[41] θ_t is about 1.5°. The

topology of the roll structure probably occurs due to the anisotropy of elastic constants in lyotropics which minimize the elastic free energy of the rolls by transferring some curvature distortion to the lowest constant-curvature mode. A similar result was observed[42] in the inversion walls of lyotropic nematics.
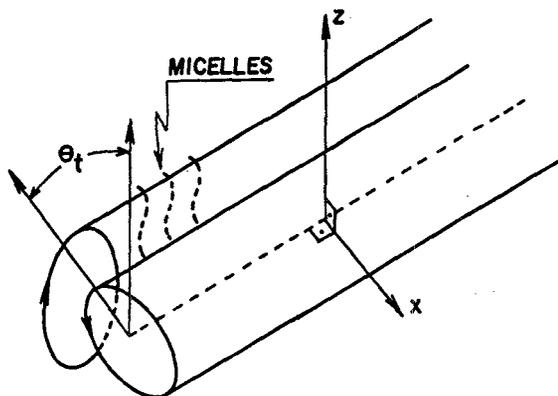
Figure 2: Sketch of the roll structure observed in lyotropic nematics.

The origin of these rolls with these features is related to the particular properties of lyotropics. One possible answer to this question could be the variation of the shape anisotropy of the micelles as a function of temperature. X-ray diffraction studies[43] indicate that, as a function of temperature, the shape anisotropy has the same behavior of the order parameter[17]. In the sample discussed in this experiment of thermal instability, at $T \approx 25°C$, the order parameter (and the shape anisotropy) is maximum and it decreases up to vanish from the nematic to isotropic phase transition when temperature increases. So, in the experimental conditions discussed, $15 < T_{co} < 42°C$ and $T_h = 44°C$, the sizes of the micelles vary along z: bigger micelles near the cold plate and smaller micelles near the hot plate. When ΔT decreases, near the hot plate, the micelles grow and need amphiphilic molecules that are picked from the bulk. This process could generate the hydrodynamic instability observed. Since the shape anisotropy of the molecules in thermotropics do not vary drastically as a function of temperature at the nematic domain, this instability has not been observed before in thermotropics.

V. **Concluding** remarks

The study of the physics of lyotropic nematics is one of the richest in both theoretical and experimental points of view. The existence of a complex phase diagram with two uniaxial and one biaxial phase also with reentrant characteristics[44] seems to be a unique one in nature. Up to now there exists considerable controversy concerning the experimental identification of a thermotropic biaxial phase. The anchoring and the thermal behavior of this phase are also very peculiar.

All the particularities of lyotropic nematic liquid crystals seem to be related to their building-blocks, i.e., the micelles, and the behavior of the micelles as a function of parameters like temperature and external torques. Differently from thermotropics, the micelles in lyotropics are not rigid objects. Far from this, the micelles are very sensitive to small changes in temperature. Their finite life-time and the possibility of changing their shape anisotropy as a function of external parameters, accessible in usual experiments are probably the motives of the unusual properties observed in lyotropics. In this scenario we hope to observe new behaviors studying the dynamical properties of lyotropic nematics. It is an open field of research for experimentalists and theoreticians in physico-chemistry with multi-disciplinar interest.

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