Final report on the project NKFI/OTKA NN 110672, entitled: Magnetically active anisotropic composite systems (MACOSYS, M-ERA.NET 2012)

The structure of the detailed final report is organized according to the key goals and the basic research questions posed by the project, as outlined in the summary.

(i.) Optical and dielectric response of ferronematics to low magnetic fields.

A thermo-stabilized setup for magneto-optical and magneto-dielectric measurements has been designed, constructed and tested. The setup is able to control/measure/collect data for the temperature, magnetic field, electric/dielectric properties and the transmitted light intensity.

The dielectric and the optical response to low magnetic fields (below 0.1T, i.e., far below the magnetic Fréedericksz threshold $B_{Fc}\approx 0.3T$) has been detected in ferronematics (FNs) based on 6CHBT nematic liquid crystal (LC) doped with spherical Fe_3O_4 nanoparticles, or with single wall carbon nanotubes functionalized with Fe_3O_4 nanoparticles (SWCNT/Fe_3O_4) in a relatively high volume concentration of 2×10^{-3} **[1]**. A similar dielectric response has been measured in a lower volume concentration range (2×10^{-4}–1×10^{-3}), and in 6CHBT doped with SWCNT, and with rod-like nanoparticles of different size and anizometry **[7]**. In the undoped 6CHBT no dielectric, nor optical response to magnetic field is detected below B_{Fc}, indicating that the addition of nanoparticles to 6CHBT increases its magnetic sensitivity considerably.

(ii.) Role of the small bias dc magnetic field and that of the anchoring at the interfaces.

One of the basic research questions of the project concerned the conditions under which the application of the small *bias magnetic field* $\mathbf{B}_{bias}$ is crucial for the optical response of ferronematics to low magnetic fields. To clarify this question, experiments have been performed with and without bias magnetic field. As a result, we have shown that the orienting bias magnetic field is not a prerequisite for the response of ferronematics to low magnetic fields. Moreover, we have demonstrated that in some cases $\mathbf{B}_{bias}$ even suppresses the response **[1]** – see, e.g., *Fig.1*.

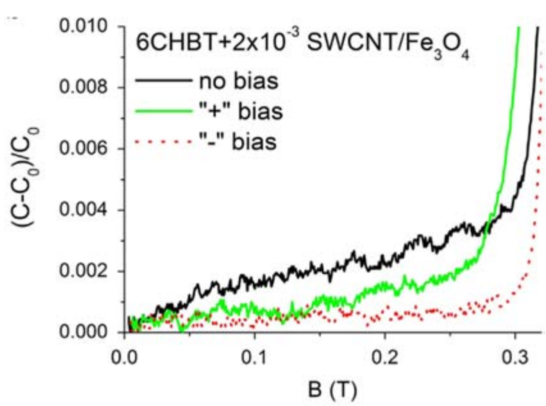


Fig. 1. The magnetic field dependence of the dielectric response (in terms of the relative capacitance) measured at T=30°C for 6CHBT doped with SWCNT/Fe_3O_4 nanoparticles.

In some other cases, however, $\mathbf{B}_{bias}$ may play an important role, and can considerably modify some physical properties of ferronematics, e.g., the *ac magnetic susceptibility*, as we have demonstrated **[12]**. Since the phenomenological explanation of this effect is associated with the aggregation/ disaggregation of the nanoparticles, we will discuss it in more details below, in **(iii.)**.

How the initial *pretilt angle* at the confining surfaces relates to $\mathbf{B}_{bias}$, and to the response of ferronematics to low magnetic fields? – it was another research question of the project. Namely, in real cells with planar orientation, the nematic director **n** encloses a small pretilt-angle with the glass plates. For cells with antiparallel rubbed polyimide layers, the pretilt-angle is typically 1° to 3°. When the reorienting magnetic field is applied perpendicular to the glass plate surfaces, the nonzero $\mathbf{B}_{bias}$ (applied parallel with the glass plates) breaks the symmetry and, therefore, one has to distinguish between "+" and "-" (opposite) directions of $\mathbf{B}_{bias}$. As we have shown **[1]**, this will cause different values of the magnetic torque exerted on **n** in the cases of B_{bias}=0, +B_{bias} ,and -B_{bias}, which will again result in different values of the threshold magnetic field for the Fréedericksz transition – see *Fig.1* around 0.3T.

We have also estimated the *anchoring strength* at the LC – nanoparticle interface from the measurements on structural transitions under the combined action of magnetic and electric fields for various ferronematics. In case of 6CHBT-based FNs doped with spherical nanoparticles a soft anchoring, while for 6CHBT doped with rod-like, or chain-like nanoparticles a rigid anchoring has been determined. The mutual orientation of the director **n** and the magnetic moment **m** of the nanoparticles has been found parallel (**m** II **n**) in all three cases **[7,P2]**. On the other hand, in ferronematics based on 6CB, a soft anchoring has been found with **m** ⊥ **n**, both with spherical and rod-like nanoparticles **[16,17]**. In general, we can conclude that both the shape of the nanoparticles and the type of the host LC are important in determining the anchoring of nematic molecules on the particle's surface.

(iii.) Influence of the aggregation on the response to low magnetic fields, and on the magnetic susceptibility.

The inevitable aggregation process of the nanoparticles in FNs has been studied from various aspects. One of these aspects was the influence of aggregation of nanoparticles on the response of the ferronematics to *low magnetic fields*. A representative example is shown in *Fig. 2*, where a sample of 6CHBT LC, and a FN cell filled with 6CHBT doped with SWCNT/Fe_3O_4 has been monitored on a long time scale. The relative capacitance versus B has been measured after different times elapsed from the cell preparation in the ferronematic system with SWCNT/Fe_3O_4, and was compared with the time independent characteristics of 6CHBT. As one sees in *Fig.2*, the first measurement on the ferronematic (made few hours after its preparation) resulted in the largest capacitive response to the applied magnetic field B. As time elapsed, the response got weaker, and within a month it almost disappeared: after 26 days from preparation, the response of the ferronematic differed from that of 6CHBT only in small details. The fact that the aggregation of nanoparticles is behind the above described effect of weakening of the response to the magnetic field was supported by the results obtained with optical microscopy: in the ferronematic sample nanoparticle aggregates of the size of tens of micrometer were observable by the end of the measurements **[1]**.

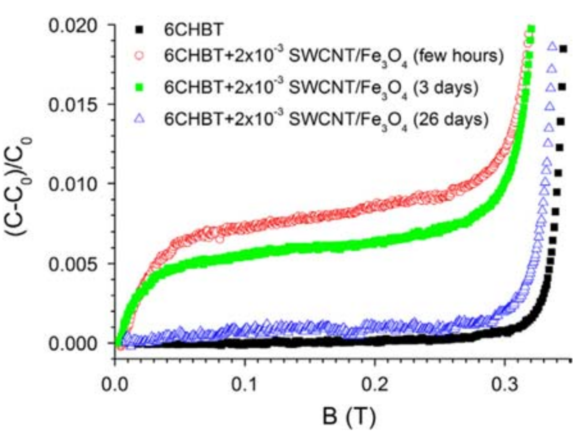


Fig. 2. The magnetic field dependence of the relative capacitance measured at T=25°C for 6CHBT and 6CHBT doped with SWCNT/Fe_3O_4 at different times elapsed from the cell preparation.

The second aspect of the investigation was to find experimentally the optimal combination of the LC host and the nanoparticle (in a reasonable concentration range of the nanoparticles), in which the composite is the most stable against the aggregation of particles. This task requires a careful selection procedure, namely, the aggregation process does not depend only on the size, shape and concentration of the particles, but for a given particle also depends on the LC matrix and on the solvent used during the preparation. For illustration, in *Fig. 3* we show a photograph of two FNs (6CHBT-based and 6CB-based), taken more than a year after their preparation (both stored at room temperature, in the nematic phase). Obviously, in the 6CHBT-based FN the particles segregated, which is clearly visible at the bottom of the container and on its wall. In contrast to that, in the 6CB-based FN no segregation is visible (neither at the bottom of the container, nor on the wall), and it has a uniform tawny color because of the dissolved nanoparticles. Under the microscope, the 6CB-based FN appears homogeneous (no particles visible), i.e., there are no aggregates larger than a micrometer. Most importantly, our most recent experimental results obtained on the 6CB-based FNs **[12,16,17]**, have been found reproducible in the course of time.

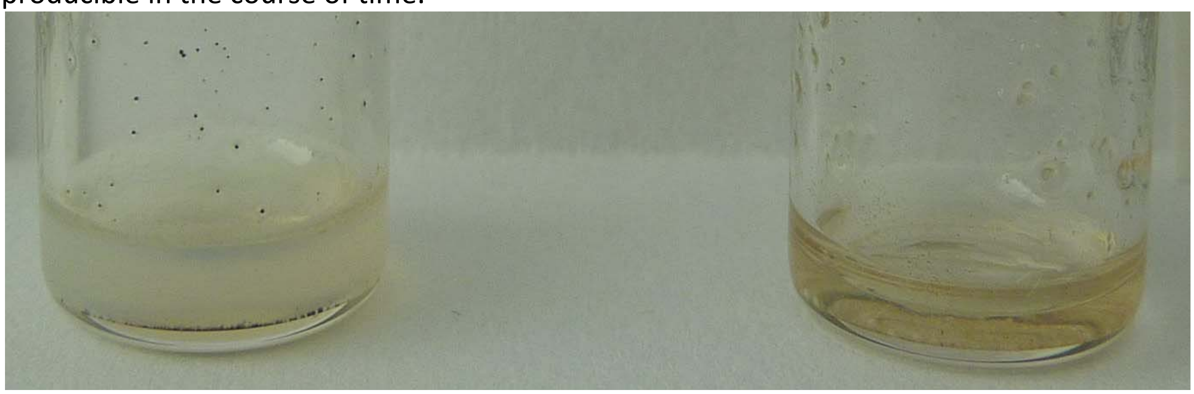


Fig. 3. Photograph made on two ferronematics prepared in the identical procedure, with the same spherical Fe_3O_4 nanoparticles (having a mean diameter of 20nm), in the same volume concentration (10^{-4}), however, in different LC matrices: 6CHBT (on the left), and 6CB (on the right). Before taking the photograph, the samples were slightly shaken, so that the FN wets the side-wall for better visibility.

The third aspect is indirectly related to the aggregation of nanoparticles, namely, the phenomenological explanation of the observed change in magnetic susceptibility of FNs involves aggregation and disaggregation of magnetic nanoparticles. The short description of the discovered effect is as follows. A small dc magnetic field (of the order of several Oe) applied in the isotropic phase increases the ac magnetic susceptibility of the ferronematic by about 10%. This enhanced value subsists while the sample is kept in the isotropic phase. Driving it through the isotropic-to-nematic phase transition resets the magnetic susceptibility to the value measured prior to the application of the dc bias field. After that, the sample could be ‘‘biased’’ again by repeated applications of the dc field in the isotropic phase only (i.e., the effect *cannot* be achieved in the nematic phase, and is *non-reversible* while passing through the isotropic-nematic-isotropic phase sequence in a cooling-heating cycle). The proposed phenomenological explanation associates the discovered effect with the aggregation of nanoparticles in the course of the isotropic-to-nematic phase transition and their disaggregation under the influence of a dc (bias) magnetic field **[12]**. Therefore, it is not surprising that the effect is inherent only to ferronematics, and it has no analogues in undoped liquid crystals, nor in water-based ferrofluids **[12]**. Although in **[12]** we presented the experimental results for a single concentration of magnetite nanoparticles in a specific liquid-crystalline matrix, the reported effect of biasing appears to be generic: our preliminary experiments on composites based on various liquid-crystalline matrices and various ferrite fillers, reveal the same effect. Moreover, the magnitude of the ac magnetic susceptibility increases with the biasing dc magnetic field only in the range of 8-10 Oe for the ferronematic reported in **[12]** (above 10 Oe the increase of the susceptibility saturates). However, our most recent (*unpublished*) experiments with a different concentration of the magnetic nanoparticles, show that the range of sensitivity to the dc bias magnetic field can easily be extended to the range of 1-100 Oe – see *Fig.4*. These findings open up the route towards real application of ferronematics as low magnetic field sensors, and to our firm belief will result in new publication(s).

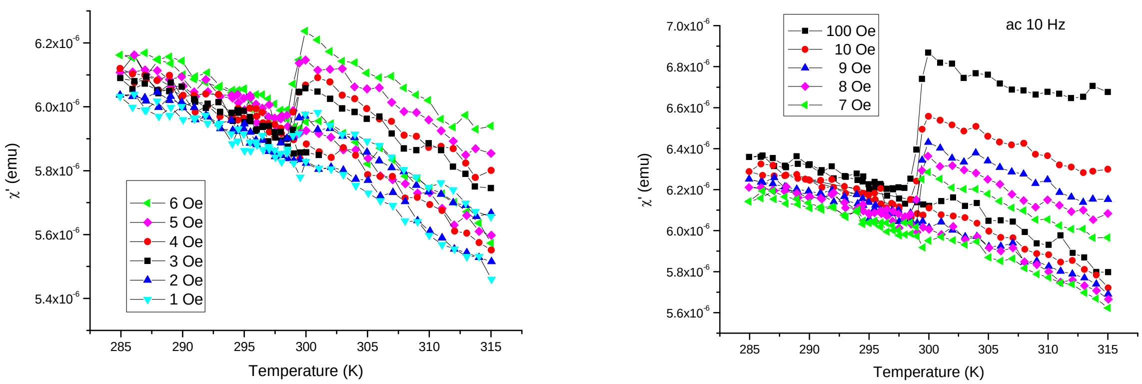


Fig. 4. Temperature dependence of the real part of the ac susceptibility of a 6CB-based FN measured at 10Hz in a cooling–heating cycle, after applying and switching off the dc bias magnetic field as indicated in the legend.

(iv.) Phase transition temperature shift induced by magnetic field and by the anisometry of the nanoparticles.

Binary mixtures composed of a calamitic and a bent-core LC have been prepared, which have been doped by spherical and rod-like magnetite nanoparticles, as well as by magnetite labeled single wall carbon nanotubes (SWCNT/Fe_3O_4). Thermographs were taken by differential scanning calorimetry for the LC mixtures **[9]**, as well as for the obtained FNs **[8]** to determine the phase sequences for all samples, and the kinetics of the phase transitions was analyzed to estimate the activation energies of the transitions.

Doping the mixture of a bent-core and a calamitic liquid crystal with spherical magnetic nanoparticles (with a mean diameter of 10nm, and in a volume concentration of 2×10^{-4}) had multiple consequences **[5]**: (A) a reduction of the critical field of the magnetic Fréedericksz transition by more than a factor of two after the doping; (B) a considerable decrease of the isotropic-to-nematic phase transition temperature T_{IN} with doping; (C) a magnetic-field-induced "negative shift" $\Delta T_{IN}=T_{IN}(B)-T_{IN}(0) < 0$ (i.e., decrease of T_{IN}). The latter observation is the first experimental evidence of the theoretically predicted magnetically induced negative shift of the phase transition temperature [Raikher *et al.*, *Soft Matter* **9**, 177 (2013)]. There is, however, a disagreement between the previous experimental findings, see e.g., **[7]** (as well as the theoretical expectations) providing a $\Delta T_{IN} \propto B^{\pm2}$ relation, and the results reported in **[5]** showing a much

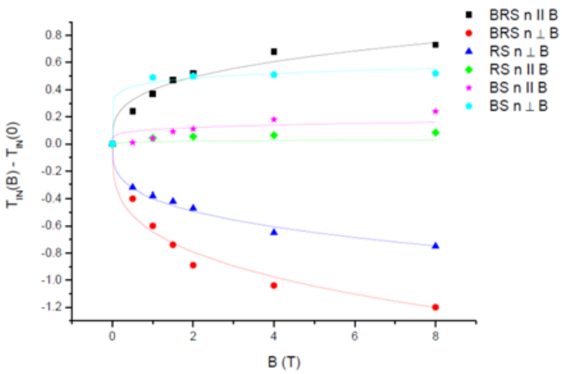


*Fig. 5. The magnetic field dependence of the phase transition temperature measured in ferronematics with calamitic (RS), bent-core (BS), and 50:50 wt% mixture (BRS) matrices, in **n**||**B** and **n**⊥ **B** geometries. The solid lines are guides to the eye.*

weaker dependence on the magnetic induction $\Delta T_{IN} \propto B^{-1/4}$. This discrepancy has triggered further, more extended systematic measurements on ferronematics formed by spherical magnetic nanoparticles with a mean diameter of 10nm, in a high volume concentration of 10^{-2}, in different LC matrices: calamitic (RS), bent-core (BS), and in their 50:50 wt% mixture (BRS), and with different orientations of the magnetic field (parallel with the initial director: **n** || **B**, and perpendicular to it: **n** ⊥ **B**). The main result are presented in *Fig. 5*, showing the magnetic field induced shifts of the phase transition temperature ΔT_{IN}. Obviously, both positive and negative shifts of have been achieved by the proper choice of the LC matrix, and of the experimental geometry. The results of these measurements are currently under evaluation, and a manuscript with provisional title *"Tuning the phase transition temperature of ferronematics with magnetic field"* is in preparation.

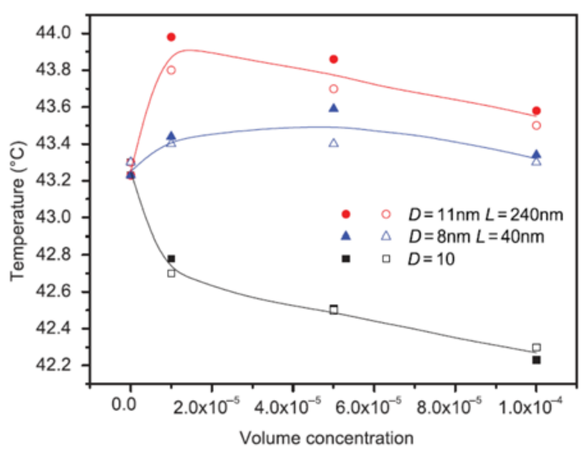


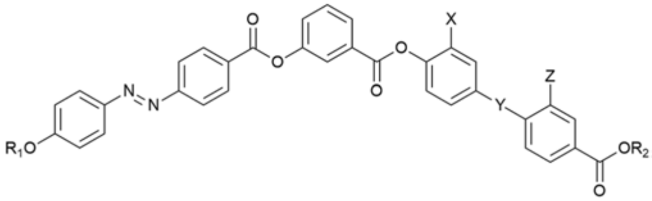
Fig. 6. Dependence of T_{IN} on the volume concentration for spherical and rod-like magnetic nanoparticles (as indicated in the legend), obtained by capacitance measurements (full symbols) and determined by polarizing microscopy (open symbols). The solid lines are guides to the eye.

We have also found that the shape as well as the volume concentration of magnetic nanoparticles have a significant influence on the temperature of the isotropic to nematic phase transition T_{IN} of ferronematics **[2]**. We have justified that ferronematics doped with rod-like magnetic nanoparticles have higher T_{IN} than the host nematic, or ferronematics containing spherical nanoparticles. We have found that doping with rod-like NPs yields a non-monotonic concentration dependence of T_{IN}, (see *Fig. 6*) which could be attributed to the competing influence of the nanoparticles and of the organic surfactants they are coated with. Our results have provided a firm experimental proof for the main conclusion of the recent mean-field theory [Gorkunov & Osipov, *Soft Matter* **7**, 4348 (2011)].

(v.) Attempts to produce magneto-, and opto-sensitive self-standing films.

Considerable efforts have been made to synthesize novel photo-sensitive LC (primarily bent-core) compounds that can be polymerized (and cross-linked) at a later stage.

First, a new series of azo-containing bent-core liquid crystals derived from 3-hydroxybenzoic acid has been synthesized (*Fig. 7*). Their mesomorphic properties have been



Compound	R_1	R_2	X	Y	Z
Ia	$C_{11}H_{23}$	C_8H_{17}	H	COO	H
Ib	$C_{11}H_{23}$	$C_{10}H_{21}$	H	COO	H
Ic	$C_{11}H_{23}$	$C_{12}H_{25}$	H	COO	H
Id	$C_{11}H_{23}$	$C_{14}H_{29}$	H	COO	H
Ie	$CH_2{=}CH(CH_2)_9$	$C_{12}H_{25}$	H	COO	H
If	$CH_2{=}CH(CH_2)_9$	$C_{14}H_{29}$	H	COO	H
Ig	$CH_2{=}CH(CH_2)_8C({=}O)$	$C_{12}H_{25}$	H	COO	H
Ih	$C_{11}H_{23}$	$C_{12}H_{25}$	Cl	COO	H
Ii	$C_{11}H_{23}$	$C_{12}H_{25}$	H	COO	Cl
Ij	$C_{11}H_{23}$	$C_{12}H_{25}$	H	–	H

Fig. 7. Chemical structures of the synthesized azo-containing bent-core LCs derived from 3-hydroxybenzoic acid.

characterized by polarizing optical microscopy, differential scanning calorimetry, small-angle X-ray diffraction and electro-optic studies **[6]**. Almost all the compounds form an enantiotropic modulated smectic (B_7 type) phase over relatively broad temperature ranges, however, at high temperatures (typically above 100°C). Structural modifications, such as the type and length of the terminal chains, the rigidity of wings, and the presence of a Cl-substituent in different positions of the bent core, affect the appearance and temperature range, but not the type of the mesophase of the investigated compounds. Light-induced changes in the texture and phase transition of the mesophase, attributed to the decrease of the order parameter due to trans–cis isomerization, have also been observed.

Second, a series of five-ring pyridine-based bent-core compounds bearing different substituents at the peripheral phenyl rings (CH_3O, Cl and NO_2) has been synthesized – see *Fig. 8*. The basic idea behind the synthesis was that pyridine itself is a highly electron deficient aromatic moiety, which can be used as the electron withdrawing part in push–pull systems. This feature opens up opportunities for obtaining photoactive materials with high quantum efficiency as well as good charge transporting ability. The mesomorphic behavior of the synthesized compounds has been investigated by polarizing optical microscopy, differential scanning calorimetry and X-ray scattering, and then compared with the unsubstituted parent compound **[13]**. The introduction of the methoxy groups at the peripheral phenyl rings of the bent core results in a non-mesomorphic compound, whereas the chloro- and nitro-substituted compounds form enantiotropic B_1-like phases (again at high temperatures, mostly above 100°C). Significant changes in the textures and in the transition temperatures of the mesophase have been observed under UV light.

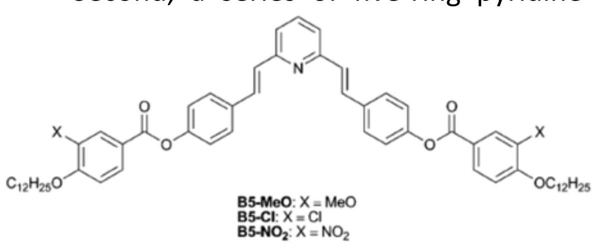


Fig. 8. Chemical structures of the synthesized five-ring pyridine-based bent-core compounds.

Third, as the above described synthetic routes has led to bent-core liquid crystals exhibiting mesomorphic properties at high temperatures, and having B_1 or B_7 LC phases, not so convenient for the preparation of magnetically sensitive self-standing films as the nematic LC is, in our focus remained the calamitic light-sensitive compound DABU synthesized at the Institute of Physics, Academy of Sciences of the Czech Republic. DABU possesses a nematic phase over a broad and convenient temperature range between 19°C and 128°C. With this novel azo-benzene-group-containing monomer several respective functional side-chain polymers grafted on a

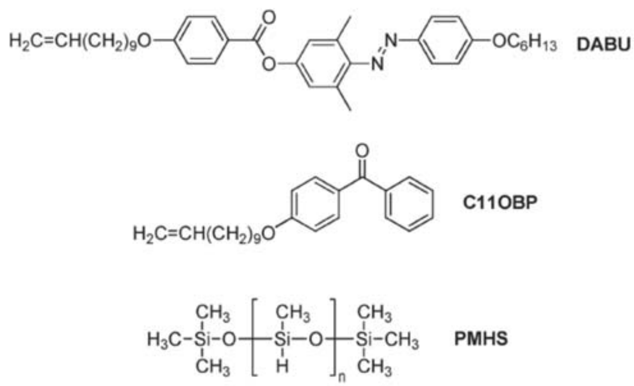


Fig. 9. Chemical structure of components used for preparation of the side-chain polymers.

methylhydrosiloxane (PMHS) backbone (with two different degrees of polymerization; with and without the addition of a photo-reactive benzophenone derivative C11OBP) were designed, synthesized, and characterized **[3]** – see *Fig. 9*. The resulting materials clearly showed self-assembly behavior and possessed a nematic LC phase over a broad temperature range, which extends below 0^oC. The optical properties of these new photochromic liquid-crystalline materials were determined from the absorbance spectra of oriented samples and by photo-induced birefringence studies. The results indicated a considerable dichroism of the sidechain liquid-crystalline polymers (SCLCPs), and hence demonstrated their potential applicability for optical storage **[3]**. Attempts have been made to crosslink the SCLCP containing the photo-reactive C11OBP with UV irradiation, however, due to the high absorption of DABU in the UV-range, the penetration depth of the irradiation turned out to be too small. The procedure resulted in a self-standing film, due to a thin outer crosslinked layer, which however, did not extend through the whole thickness of the film, and therefore, it was immediately destroyed upon any excitation.

(vi.) Periodically distorted states of nematics.

Though no influence of the periodically distorted state of the nematic liquid crystal to the aggregation process has been measured (primarily because aggregates sedimented on the orienting polymeric surface, and could not be removed/disaggregated by the electro-convective flow), significant contributions have been made to the research of the electro-convective and flexo-electric pattern forming processes in nematics.

As either ac or dc voltages can induce periodically distorted states in nematics, one expects that patterns will appear also at superposed ac and dc driving. Two nematics with different dc behaviour were selected for the investigations; one (Phase5) exhibited non-dissipative flexodomains (FDs), the other (1OO8) showed dissipative electroconvection (EC) at dc driving. The onset characteristics (threshold voltages and critical wave vectors) were determined in a wide frequency range of the ac voltage, including both the conductive and the dielectric regimes. In contrast to our expectation and the predictions of the standard theory of electroconvection (EC), our experiments showed that, in general, the superposition of driving with different time symmetries (ac + dc) rather inhibits pattern formation than favors the emergence of instabilities; thus the pattern free region may extend to much higher voltages than the individual ac or dc thresholds **[4,14]**. Some examples for the stability limiting curves under superposition are shown in *Fig. 10*. The combined driving usually causes morphological transitions when moving along the stability limiting curve in the ac-dc plane. These include crossover from conductive EC to dielectric EC, from EC to FD, or a peculiar transition between two types of flexodomains with different wavelengths [FD and FDSW, *Fig.10(b)*].

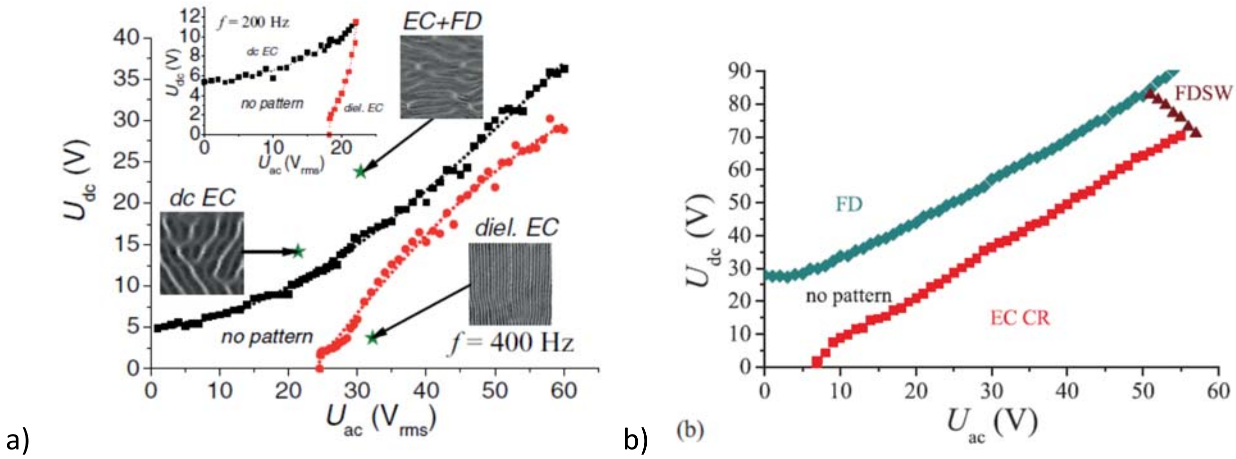


Fig. 10. Morphological phase diagram under superposed ac and dc voltages. a) Combination of dc EC with ac dielectric EC in Phase 5, f = 400 Hz (inset: f = 200 Hz); b) combination of dc FDs with ac conductive EC in 1OO8, at f = 5 Hz.

We proved that the dielectric permittivity is not affected by a dc bias; however, the conductivities as well as the relative conductivity anisotropy decreases substantially upon the dc bias voltage **[14,P3]**. We showed that taking into account the experimentally detected variations of the conductivity in the linear stability analysis of the underlying nematohydrodynamic equations, a qualitative agreement with the experimental findings on the onset behavior of spatially periodic instabilities could be obtained **[14]**.

Electric-field-induced patterns of diverse morphology were observed over a wide frequency range in a recently synthesized oxadiazole bent-core nematic (BCN) liquid crystal (9P-CF_2O-ODBP). At low frequencies (below 25 Hz), the BCN exhibited unusual polarity-dependent patterns. At higher voltage amplitude, these time-asymmetrical oblique roll patterns turned into time-symmetrical prewavy-like stripes. At higher frequencies, zigzag and/or prewavy-like patterns were detected **[11]**. All these pattern morphologies belong to nonstandard electroconvection; their interpretation would be a challenging theoretical task.

In a shorter-chain member (7P-CF_2O-ODBP) of the same homologous series, regular domain structure consisting of parallel stripes – flexodomains – were induced by low frequency (sub-Hz) or dc electric voltages. The FD pattern serves as an optical grating tunable with the voltage and produces a regular system of laser diffraction spots. The polarization of the first order diffracted light spot was found perpendicular to that of the incident light **[10]**.

A unique feature of FDs is that their wavelength depends linearly on the amplitude of the applied voltage, which makes them suitable for beam steering applications. The dynamics of the switching process was also studied, proving that the system responds to increasing voltage levels much slower than to decreasing voltage levels **[18]**.

As an appreciation of their decade-long research efforts and results, project participants were invited to prepare a comprehensive review on the electric field-induced patterns in liquid crystals. The review appeared in a high impact international journal **[15]**.

Publications in peer-reviewed international journals (with the supporting NKFI grant number indicated):

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