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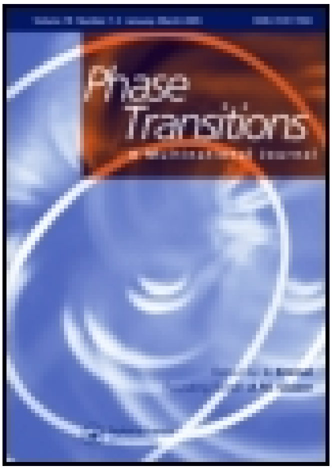

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Electro-optic properties of nematic and ferroelectric liquid crystalline nanocolloids doped with partially reduced graphene oxide

Valeri Lapanik[a], Sergei Timofeev[a] & Wolfgang Haase[b]

[a] Laboratory of Condensed Matter Optics, Institute of Applied Physics Problems, Belarusian State University, Minsk, Belarus

[b] Condensed Matter Research Group, Eduard-Zintl-Institute for Inorganic and Physical Chemistry, Technical University Darmstadt, Darmstadt, Germany

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Electro-optic properties of nematic and ferroelectric liquid crystalline nanocolloids doped with partially reduced graphene oxide

Valeri Lapanik[a]*, Sergei Timofeev[a] and Wolfgang Haase[b]

[a]*Laboratory of Condensed Matter Optics, Institute of Applied Physics Problems, Belarusian State University, Minsk, Belarus;* [b]*Condensed Matter Research Group, Eduard-Zintl-Institute for Inorganic and Physical Chemistry, Technical University Darmstadt, Darmstadt, Germany*

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Flakes of partially reduced graphene oxide (PRGO) were doped in nematic liquid crystals (NLCs) and ferroelectric liquid crystals (FLCs), respectively. The dielectric and electro-optical properties of NLCs doped with those flakes have been investigated. Threshold voltage and switching times are reduced by 30%−50%. This is primarily due to the decrease of the elastic properties of the nanocolloids compared to the non-doped nematics. The influence of the PRGO flakes on the spontaneous polarization, tilt angle and switching time of FLCs was investigated too. Such flakes reduce the response time by 40%−60%, increases spontaneous polarization by 20%−25% and increase the tilt angle by 15%−20%.

Keywords: partially reduced graphene flakes; nematic and ferroelectric liquid crystals; electro-optical response; elastic properties

1. Introduction

Development of novel liquid crystal (LC) composite materials represents a new direction in materials science. LC material is in general a multi-component mixture consisting of compounds of different chemical classes. The optimal set of physical-chemical parameters (temperatures of phase transitions, viscosity, dielectric and optical anisotropy, etc.), which define electro-optical characteristics of display devices, is achieved by the selection of individual components of the mixture and their amount.

In recent years, the attention of researchers was focused on the modification of properties of LCs due to doping with various nanoparticles in order to obtain enhanced electro-optical characteristics. Compositions of LCs with metallic, semiconducting [1−3] as well as ferroelectric [4−6] nanoparticles have been investigated. It has been shown that a small amount of nanoparticles to a great extent modifies dielectric, elastic and electro-optical properties of LCs.

The growing interest in carbon-based materials is associated with their effective application in various directions. In this regard, the combination of LCs and synthetic carbon allotropes such as carbon nanotubes (CNTs), detonation nanodiamonds (DNDs) [7] and the newly described graphene has its own importance.[8−10]

CNTs bring in novel properties that make them potentially useful in a wide range of applications in nanotechnology, electronics, optics and other fields of materials science. CNTs possess extremely high aspect ratio with diameters of the order of nanometers and length larger than several hundred nanometers. Dispersing CNTs in nematic liquid

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*Corresponding author. Email: lapanik@bsu.by crystals (NLCs) and ferroelectric liquid crystals (FLCs) [11] is one of the possibilities to modify and improve the physical–chemical and electro-optical properties of LCs useful for display devices. It was found that the introduction of CNTs in nematic LCs as a dopant can depress the undesired ion-charge effect [12–15] and simultaneously reduce the rotational viscosity [16] of the LC host, and, as a consequence, decrease the response time, suppress the backflow effect and decrease the threshold voltage.[12,17] But even an increase of the viscosity by increasing the amount of CNT for a certain FLC mixture has been reported.[18]

But not only tubes like CNTs with large aspect ratio are of great interest, e.g. for screening ions, but also rods, for example gold nanorods (GNRs). Moreover, the influence of the diameter of GNRs on the electro-optical and dielectric properties of FLCs has been investigated.[19]

Among the various synthesized carbon structures, it is necessary to note the DNDs obtained by the method of detonation synthesis. Such DNDs are aggregates of diamond single crystals with an average size of 3–6 nm. Unlike the CNTs, DNDs have a spherical shape. A distinctive feature of the DNDs is the presence of a large number of different functional groups on their surface. In [7], the impact of the DND nanoparticles' size and the type of functional groups on the dielectric and electro-optical properties of nematic mixtures was studied. It was shown that nanoparticles of small size have virtually no effect on the properties of LCs. At the same time, the conglomerates based on DNDs of about 50 nm or about 100 nm in diameter can increase or decrease the dielectric anisotropy and response times by 1.5–2 times, depending on the polarity of the functional groups.

The other allotrope of carbon, graphene, is a newly promising material for nanoelectronics. It exists in standard samples in quasi-two-dimensional isolated monolayers of carbon atoms arranged in a hexagonal lattice. Graphene exhibits exceptional mechanical, optical, electronic and thermal properties including high charge mobility, thermal conductivity, high strength, high chemical stability and high optical transmittance.

Several physical and chemical methods exist for the synthesis of graphene, which include the mechanical or chemical exfoliation of graphite, unrolling of CNTs (either through electrochemical, chemical or physical methods), chemical vapor deposition, reduction of graphene oxide (GO), etc.[20–24]

Potential graphene applications include carbon-based nanoelectronics, biomedical engineering, ultrafiltration, composite materials, photovoltaics and energy storage. The high electrical conductivity and high optical transparency promote graphene as a candidate for transparent conducting electrodes, required for applications in touch-screens, LC displays, organic photovoltaic cells and organic light-emitting diodes.[25–30]

Studies of LC-graphene or GO composites dispersed in the LCs are to date rather rare. For the nematic mixture E7, an increase of the threshold voltage has been reported [31] while for graphene-5CB nanocolloids, a decrease of the orientational order was described by the use of ^{2}H NMR methods.[32] Very recently, decrease of switching time and decrease of rotational viscosity has been reported for the system graphene-5CB nanocolloid.[33] On the other hand, for the zero-dimensional derivative of graphene, mean for graphene quantum dots, improvement of the electro-optical properties and reduction of the threshold voltage have been described for the nematic nanocolloids.[34] Investigation of graphene doped in FLCs has been reported very recently. A remarkable decrease of the rotational viscosity has been described by Basu.[35] For the GO, the Biradar group [36] reported via measurements with help of the automatic LC tester (ALCT, Instec) on an increase of the viscosity for an electroclinic FLC-graphene nanocolloid in comparison to the non-doped FLC.

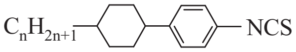
The aim of our work is to describe the electro-optic and dielectric properties of nanocolloid dispersions of partially reduced graphene oxide (PRGO) in some nematic and ferroelectric LCs, respectively, and to compare those with properties of LC nanocolloids of graphene and of PRGO.

2. Materials and methods

PRGO is a gift of Dubang China Group Co., Ltd. It has been used without further purifications. For the characterization of the PRGO, scanning electron microscopic investigations by means of LEO-1455 VP SEM has been performed. This microscope was equipped with energy-dispersive X-ray spectrometer (EDX) with RONTEG analyzer. Each powder sample was placed on an aluminum spot with double-sided carbon tape. The voltage for the energy dispersive analysis was 20 keV.

The powder consists of flakes of thickness <5 nm, formed by monolayers and oligolayers. The flakes show dimensions with longer <1000 nm and shorter diameter <250 nm. The compositions after SEM/EDX are C 87.10 wt% and O 10.90 wt %, rest are H and few metallic impurities. It should be mentioned here, in GO the ratio C:O is usually 2.1:1−2.9:1 while in our case C:O ~ 9:1.[37]

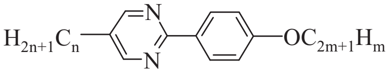
For the experiments with NLC, a host mixture **N** based on the 1-alkyl-4-(4′-isocyanatophenyl)-cyclohexane



has been prepared. **N**: $n = 3$: 27.0 wt%; $n = 4$: 19.0 wt%; $n = 6$: 37.0 wt%; $n = 7$: 17.0 wt%.

The phase diagram of the mixture **N** is Cr 12 °C N 43.2 °C I.

FLC mixtures were prepared based on substituted 2-(4’-alkoxyphenyl)-4-alkyl-pyrimidines



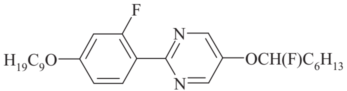
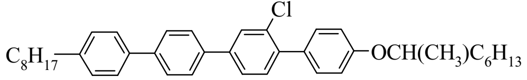
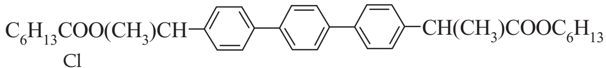
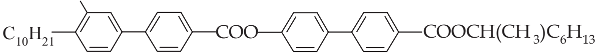
forming the SmC phase of the mixture **M.**

M: $n = 10$, $m = 8$: 43.3 wt%; $n = 8$, $m = 8$: 23.4 wt%; $n = 8$, $m = 10$: 33.3 wt%.

The phase diagram of the mixture **M** is Cr 7 °C SmC 57.8 °C SmA 67.2 °C N 68.1 °C I.

In order to prepare a mixture with a ferroelectric C* phase, we added to the host mixture **M** different amount of chiral dopants $\mathbf{A_1}$ to $\mathbf{A_4}$ (Table 1) leading to the FLC mixtures

Table 1. Chiral compounds used for FLC mixtures **F-1** to **F-4**.

	Chiral compounds
$\mathbf{A_1}$	
$\mathbf{A_2}$	
$\mathbf{A_3}$	
$\mathbf{A_4}$	

F-1 (M + 15% A_1), F-2 (M + 25% A_2), F-3 (M + 25% A_3) and **F-4 (M + 25% A_4)**. Chiral compounds of different viscosity and with different functional chiral groups were selected to study their influence on the spontaneous polarization and the elastic properties.

PRGO was dispersed in propanol through ultrasonic bath for 1.5 h. Then, the dispersion was mixed with NLCs and FLCs, respectively. In detail, the nematic mixtures **N** were transformed into **GN** due to the doping with PRGO 0.5 wt%, while to the four FLC mixtures **F-1** to **F-4** each 0.5 wt% of PRGO were added leading to the doped mixtures **GF-1** to **GF-4**.

The obtained mixtures were inserted inside the oven at 70 °C for 0.5 h and then degassed under vacuum (1−3 mbar) for 0.5 h in order to evaporate propanol.

The compositions prepared in such a way were filled in the cells with a thickness of 3.5 μm for NLC and 2.0 μm for FLC. Planar and 90°-twist cells with an effective indium tin oxide (ITO) area 1 cm × 1 cm have been used. The quality of LC alignment and the sample morphology were controlled using polarizing optical microscope (Olympus BX-51P) interfaced with charge-coupled device (CCD) camera at 10x in the transmission mode in crossed polarizers using white light. Electro-optical parameters were measured using the setup consisting of a He−Ne laser ($\lambda = 632.8$ nm), rotating table, Linkam LTS 350 hot stage coupled with the temperature controller Linkam CI 94 (with an accuracy ±0.1 °C), arbitrary waveform generator HP 33120A and the digital oscilloscope HP Infinium. The rise times were defined as the transmittance changes from 10% to 90%. The light transmittance was detected by a photomultiplier and recorded via a digital oscilloscope. Spontaneous polarization (P_s) was measured using the reversal current method with a triangular wave at 10 Hz obtained from HP 34401A. The amplitude of the applied voltage was 20 V_{pp}. The tilt angle was estimated from the measurements of light transmission through the cells in crossed polarizers under applied electric field. The transmission was fitted as a function of the cell orientation to the sinusoidal curve. Dielectric measurements were carried out by impedance analyzer 4192A at frequency 1 kHz. In order to measure the elastic constant K_{11} of the nematic LC mixtures, the standard method was used based on the investigation of Freedericksz transition.[38] Freedericksz transition was induced by electric field and detected optically. The thickness of the empty cells was measured by the interferometric method.

3. Results and discussion

One of the aims of the research of pure nematic mixtures **N** in comparison with PRGO-doped mixtures **GN** was to study the changes of certain physical properties due to doping. Consideration of model of interaction between PRGO and nematic mixtures was another goal.

In order to determine the elastic constants K_{11} of the doped and non-doped NLC mixtures, the phase difference as a function of voltage were measured in planar cells. From the threshold voltage, elastic constants were calculated

$$K_{11} = \frac{U_{th}^2 \cdot \varepsilon_0 \cdot |\Delta\varepsilon|}{\pi^2} \tag{1}$$

where K_{11} is splay elastic modulus, $\Delta\varepsilon = \varepsilon_{||}\text{-}\varepsilon_{\perp}$ − dielectric anisotropy, and $\varepsilon_0 = 8.854 \cdot 10^{-12}$ farad/m.

For the pure nematic mixture, the value of $K_{11} = 6.9$ pN, for the doped one $K_{11} = 5.1$ pN.

Figure 1 presents the transmission−voltage curve measured in a 90°-twist cell for both non-doped NLC and PRGO-doped material.

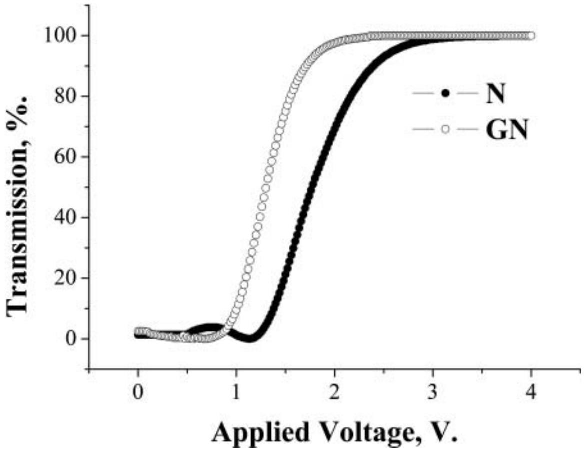


Figure 1. Voltage-transmission curve for non-doped NLC and NLC doped with PRGO at 25 °C.

The optical threshold voltage V_{10} has been reduced for the doped case. This corresponds to a reduction of elastic modulus of the doped nematic, whereas the magnitude of the dielectric anisotropy has not changed, as will be shown below.

In the following experiment (Figure 2), we determined the frequency dependence of dielectric anisotropy. The measured parallel and perpendicular dielectric data have been presented as a function of the applied frequency both for the non-doped and the doped material.

The dielectric anisotropy has not been changed due to doping with 0.5 wt% PRGO.

Figure 3 shows the decrease of the rise time τ_r from about 1 ms for the non-doped case to about 0.5 ms for the doped case.

In the planar alignment, the nematic molecules are oriented parallel to the sample surface. We suppose the PRGO plates are parallel to the nematic molecules disturbing the

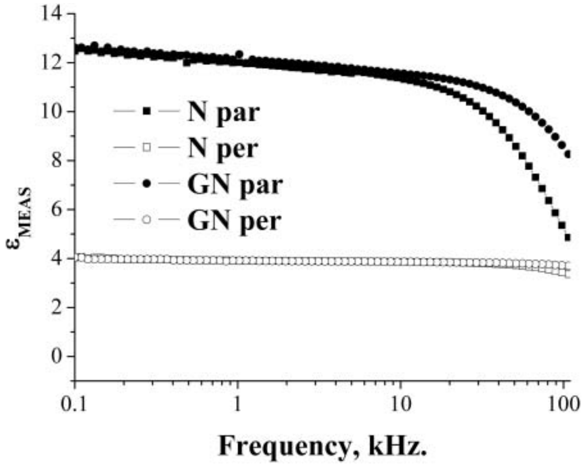


Figure 2. Frequency dependence of dielectric permittivity for non-doped NLC and NLC doped with PRGO at 25 °C.

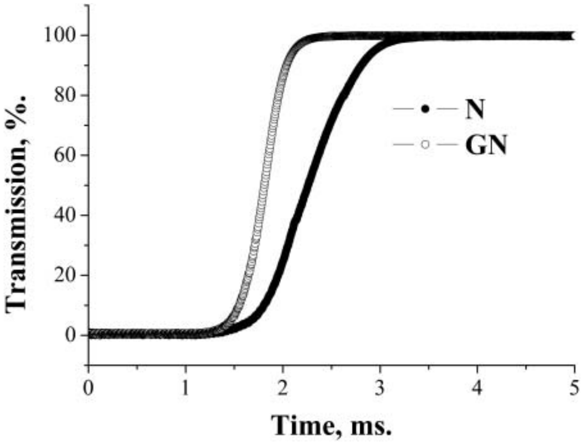


Figure 3. Time response for non-doped NLC and NLC doped with PRGO at 25 °C.

short-range ordering between them. Moreover, there might be some $\pi - \pi$ interaction between the PRGO plates and the nematic molecules leading to the weakening of LC intermolecular interactions.

All findings obtained so far are very interesting for potential applications, because the rise time dropped and the threshold voltage was lowered.

The influence of PRGO-flakes on the properties of FLCs is even more interesting. In Figure 4, we present the frequency dependence of the real (a) and the imaginary (b) part of the dielectric permittivity.

Due to doping with PRGO, both absolute values up to about 30 kHz were reduced. One can assume the PRGO flakes are able to compensate and neutralize some ions or charges present inside the FLC volume leading to the decrease of both the dispersive and absorptive values at lower frequencies. In both diagrams (Figure 4), the frequency of the Goldstone mode (at about 1 MHz the so-called ITO-mode appeared) became slightly shifted to higher values. The frequency of the Goldstone mode f_G is proportional to the (Goldstone mode) elastic constant $K_{\rho,}$ and the second power of the wave vector q, but

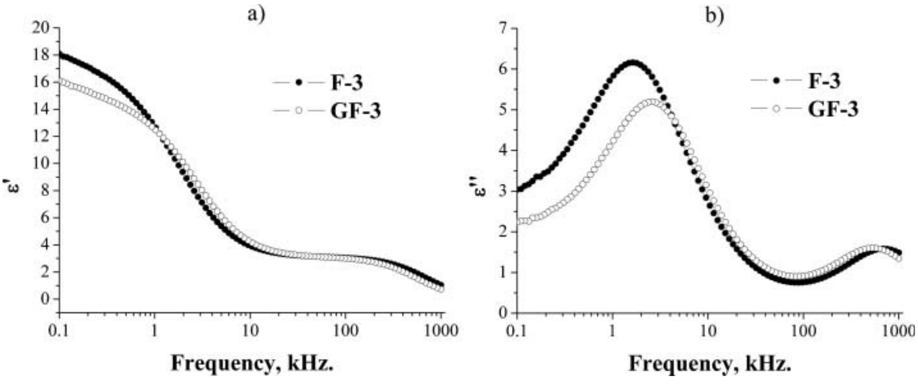


Figure 4. Frequency dependence of (a) dielectric permittivity and (b) absorption strength at 25 °C.

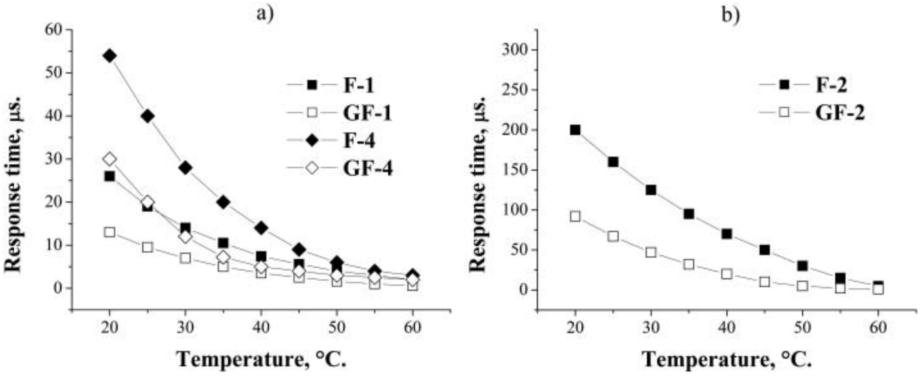


Figure 5. Temperature dependence of the response time of non-doped FLC and FLC doped with PRGO.

inversely proportional to the Goldstone mode viscosity γ_G which can be assumed as the rotational viscosity,[39] $f_G = K_\rho q^2/2\pi \gamma_G$.

One can assume the slight increase of the Goldstone mode frequency is due to the decrease of the viscosity assuming the wave vector and elastic constants became constant. In practice, the elastic constant as shown for the nematics is being reduced but the wave vector due to doping gets slightly increased as has been shown recently.[40] As a model, the increase of Goldstone frequency is primarily due to decreased viscosity while decreased elastic constant and increased wave vector might compensate each other in their influence.

Figure 5 shows the decrease of the response time by increasing the temperature for the doped mixtures **GF-1**, **GF-2** and **GF-4**, respectively, compared to the related non-doped ones. This decrease of response time while doping can be compared with the decrease in the switching time of the nematics.

By the same time, the spontaneous polarization (Figure 6a **F-3, GF-3;** Figure 6b **F-2, GF-2; F-4, GF-4**) and the tilt angle (Figure 7 **F-1, GF-1; F-3, GF-3**) were both increased (Table 2).

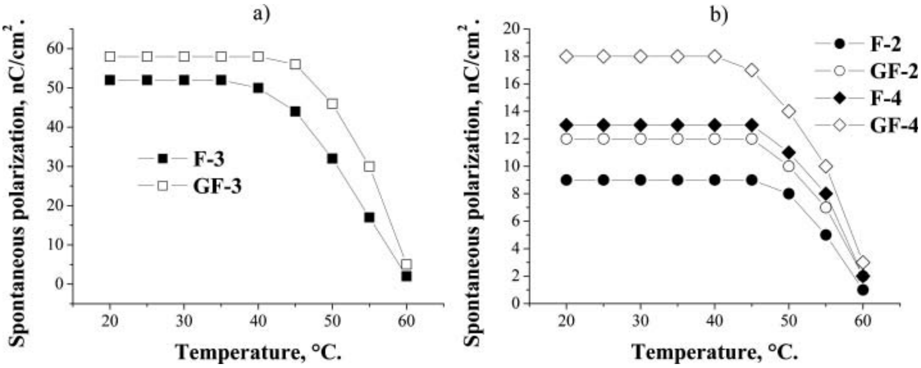


Figure 6. Temperature dependence of the spontaneous polarization of some non-doped FLCs and related FLCs doped with PRGO.

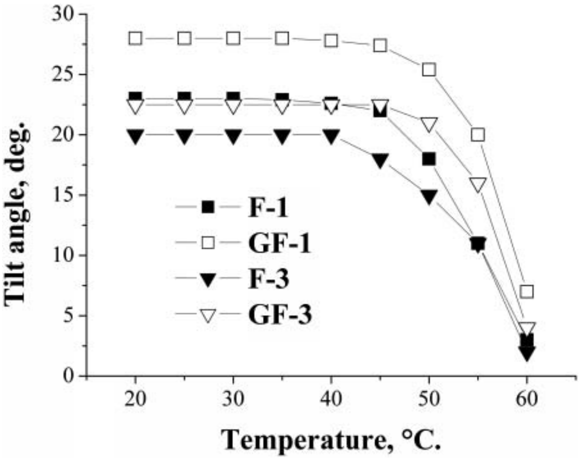


Figure 7. Temperature dependence of the tilt angle of some non-doped FLCs and related FLCs doped with PRGO.

The different chiral dopants $\mathbf{A_1}$ to $\mathbf{A_4}$, characterized by different values of the helical twisting power and spontaneous polarization, contribute to the increase of the tilt angle and spontaneous polarization of mixtures, and as a consequence, to the reduction of switching time.

When building a model of the FLCs doped with PRGO, one must take several aspects into consideration.

The smaller-sized PRGO plates are packed parallel to the chiral and the host molecules of the FLC matrix inside the smectic layers leading to $\pi-\pi$ interaction between PRGOs and benzene groups of the FLCs. As a result of these interactions, the ordering increases forming a coupling of the dipoles of PRGO and FLC in a parallel manner which even leads to the increase in the spontaneous polarization. Due to the decrease in viscosity, the switching time became reduced. Compared to graphene, the PRGOs plates might be not as planar showing some staggering. In a very recent report, [35] some enhanced smectic C-domains were postulated. We do not see such domain forming in our FLC-PRGO-nanocolloid. Regardless of these electro-optic properties of PRGO and graphene, FLC-nanocolloids are similar in tendency. For GO-FLC nanocolloid, viscosity increases while doping.[36] This is different to PRGO.

Table 2. Physical and electro-optical parameters of FLCs.

Mixture	P_s, nC/cm^2	Tilt angle, °	τ, μs
F-1	10	23	22.0
GF-1	13	28	12.0
F-2	9	23	220
GF-2	12	27.5	92
F-3	52	20	9.8
GF-3	58	22.5	7.2
F-4	13	23	54.0
GF-4	18	25	28.0

Finally, it is well known that dopants with bigger-sized platelets dispersed in LCs tend to organize themselves after ultrasonicating parallel to the polymer/ITO surface area that leads to a sort of demixing. Unfortunately, we do not have access in use of HRTEM or SEM to control sizes and organizations inside the cell. One even cannot exclude that larger-sized platelets became partially crushed via ultrasonification.

4. Conclusions

It is established that the effect of graphene or GO on the physical and electro-optical parameters of NLC and FLC is very strong. Here, we tried to describe a material between graphene and its oxidized form, GO, by considering the PRGO. The introduction of PRGO in the nematic matrix can significantly affect the viscous-elastic properties of the composition, and especially change the elastic constants, which reduces the switching times and the threshold voltage. As expected, PRGO has no effect on the value of the dielectric anisotropy, since it has no permanent dipole moment.

As for FLCs, the small amount of dispersed PRGO plates leads to the increase of the tilt angle and the spontaneous polarization. At the same time, the interaction energy between the FLC molecules has been reduced because they became separated due to the PRGO flakes leading to the reduction of the switching time. In general, the PRGO plates act as an ion/charges scavenger in the LCs which can be seen in the change of the impedance spectra at lower frequencies.

In general, the electro-optic properties of PRGO with the ratio C:O $\sim$ 9:1 can be compared to those of graphene while some similarities to GO with the ratio C:O $\sim$ 2.1:1–2.9:1 exist.

Disclosure statement

No potential conflict of interest was reported by the authors.

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