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Influence of Goethite Nanorods on Structural Changes and Transitions in Nematic Liquid Crystal E7

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Abstract: A composite ferronematic system based on the nematic liquid crystal E7, doped with lath-like goethite magnetic nanoparticles of volume concentrations 10^{-3}, 5×10^{-4}, and 10^{-5}, was investigated. Both surface acoustic waves (SAWs) and the magneto-optical effect were used to study the influence of magnetic nanoparticles on ferronematic liquid crystals' structural changes, focused above all on structural transitions. The responses of SAW attenuation and light transmission to external magnetic fields were investigated experimentally under linearly increasing/decreasing or jumped (time influence) magnetic fields, respectively. An investigation of temperature on structural changes was performed, as well. The experimental results validated the decrease in the threshold field of the ferronematic composites in comparison with the pure E7, as well as an increase in the transition temperature with the increasing volume fraction of nanoparticles. The effect of the nanoparticles' concentration on both total structural changes and residual attenuations at the vanishing magnetic field was also registered. The light transmission measurements confirmed the effect of the concentration of goethite nanoparticles on the resultant magneto-optical behavior, concerning both its stability and switching time.

Keywords: liquid crystals; ferronematics; goethite nanoparticles; structural transitions

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1. Introduction

The anisotropy of nematic liquid crystals (NLCs), combined with particular properties of nanoparticles, generates possibilities for the development of materials with the perspective of nanotechnological progress, particularly in nanoscale electronics, sensors, display devices, and electro- and magneto-optics [1,2]. NLCs doped with magnetic nanoparticles (ferronematics) are magnetically active fluids, which are a manifestation of the initial idea of [3], supposing that the immixing of fine magnetic particles in liquid crystals may improve their sensitivity to magnetic fields. Liquid–crystalline composites compromising mineral particles are a special category of fluid suspensions due to not only their anisotropy, but also the special magnetic and transport properties of these compounds [4]. At present, considerably more interest is paid to mineral LCs, which are represented as colloidal suspensions of goethite particles in solvents than to organic LCs. These compounds have some enhanced properties, including optical, electrical, and magnetic ones. Better thermal stability is another important factor. LCs with goethite (α-FeOOH) are a kind of mineral LC that is extremely sensitive to external magnetic fields. Goethite represents a ferric compound that has been investigated for many applications due to its chemical stability at room temperature [5–7]. Goethite nanoparticles are characterized by permanent magnetic moment along the particle's long axis, whereby they show different magnetic properties compared with bulk materials. Then, they can orient in a low-intensity magnetic field parallel to the field [8,9]. The most important properties for monitoring LC behavior under magnetic fields are the threshold behavior in the re-orientational response of LCs, characterized by director ***n*** and the nematic–isotropic temperature shift.

Based on the results of a theoretical paper describing the behavior of magnetic nanoparticles in liquid crystals [10], the shape of nanoparticles plays an important role in influencing nematic–isotropic phase transition temperatures. While spherical nanoparticles reduce the temperature of the isotropic–nematic phase transition, anisotropic nanoparticles raise it. The results of experimental studies [11–13] demonstrate that doping with spherical iron oxide nanoparticles results in a lowering of the isotropic–nematic phase transition temperature, while nanorod-shaped particles result in a shift toward a higher temperature [14,15]. The effect was, in both cases, intensified by increasing the concentration [11].

Concerning the threshold field, most magnetic nanoparticles have been found to be able to reduce a threshold magnetic field [12–17]. Although many experimental works have reported decreases in threshold magnetic fields in composites of various LCs and magnetic nanoparticles, an increasing threshold of magnetic fields can occur, as well [11,18]. A threshold magnetic field is reduced or increased depending on the mutual orientation of the LC director and the nanoparticle's magnetization vector. A threshold magnetic field applied perpendicularly to an LC molecule with a nanoparticle magnetization vector parallel to the director decreases because the magnetic nanoparticles assist in LC molecule rotation via coupling between nanoparticles and LC molecules. Because the LC molecules must also overcome the coupling between nanoparticles and LC molecules when the initial orientation of the LC director is not parallel to the nanoparticle's magnetization vector, the threshold magnetic field increases. In general, nanoparticles have an impact based on their properties, which include the right materials, sizes, shapes, and concentrations of nanoparticles.

The present paper partly continues the study of goethite nanorods' influence on structural transitions in LCs, previously studied using dielectric measurements and monitored using polarizing microscope [19] on goethite-doped 6CHBT. By adding goethite nanorods to LCs, we demonstrated that they would affect the LCs' sensitivity to external magnetic fields, reduce phase transition temperatures, and reduce threshold fields, which can be further reduced by doping them with goethite magnetic nanoparticles. The aim of the paper is to verify the influence of goethite nanorod particles on both threshold field and nematic–isotropic transition temperatures with the intention of structural changes cognition in E7 composites, investigating the responses of SAW attenuation and light transmission.

2. Experiment

2.1. Materials

The ferronematic LC sample was based on the nematic E7, which is composed of four liquid crystals—5CB, 4-cyano-4′-n-heptyl-biphenyl (7CB), 4-cyano-4′-n-oxyoctyl-biphenyl (8OCB), and 4-cyano-4″-n-pentyl-terphenyl (5CT), which is an LC with high chemical stability [20]. E7 was doped with goethite magnetic nanoparticles of volume concentrations 10^{-3}, 5×10^{-4}, and 10^{-4}. The synthesis of goethite nanorods was provided by Fe $(NO_3)_3 \cdot 9H_2O$ (99% pure) as a metal oxide precursor, NaOH for the preparation of the co-precipitation solution, and deionized water as a solvent and base fluid for nanopowder preparation. Goethite nanorods were prepared by adding 5 mL of a $Fe(NO_3)_3$ 0.06 M solution dropwise to 45 mL of a NaOH 1.5 M solution at room temperature, leading to a ferrihydrite precipitate. The red precipitate was aged in a mother liquor solution for three days leading to a yellow precipitate of goethite nanorods. The goethite precipitate was washed by centrifugation several times with deionized water. The pH, 11, was ensured with a NaOH solution. Firstly, the composite with volume concentration 10^{-3} was prepared and samples with another volume concentration, 10^{-4} and 5×10^{-4}, were worked off by mixing in an additional amount of E7. Stable suspensions of goethite nanorods into the LC were obtained by the procedure that has been already described [19]. A mean length of 350 ± 100 nm, width of 25 ± 7 nm, and thickness of 10 ± 5 nm for goethite nanorods were estimated using a Sigma Zeiss field emission SEM operating at 10 kV.

2.2. SAW Measurements

The liquid crystal cells of thickness $D \approx 100$ µm of investigated compounds were situated on the $LiNbO_3$ substrate at the center. Two interdigital transducers were prepared near edges of the $LiNbO_3$ delay line. The $LiNbO_3$ substrate was installed in the sample holder that was insert into the thermostatic measuring chamber. The first interdigital transducer generated SAW pulses of frequency 10 MHz (~1 µs) using hf pulses, and the second transducer was used as receiver of SAW signal, both using the pulse generator and receiver of MATEC 7700. The SAW attenuation response was recorded by MATEC Attenuation Recorder 2470. In SAW attenuation measurement, it is supposed that the starting internal LC molecules arrangement should have a predominate alignment in the plane of LC cell. The magnetic field is applied perpendicular to this orientation. The external magnetic field reorients LC molecules to the direction perpendicular concerning the cell surface, starting at the center of LC cell layer, due to the bonding between magnetic moments of LC molecules and magnetic nanoparticles. The amplitude of SAW reflects the fact that, after reaching a LC layer, the SAW radiates a longitudinal wave into LC [12] giving rise to the propagation losses. The SAW attenuation α by this way can be responding to any changes in LC orientational changes induced by external terms (temperature, illumination, magnetic and electric fields, . . .). The complete experimental arrangement configuration has been already described [15,21]. The instability for $\Delta\alpha$ measurement was smaller than ±0.02 dB. The temperature in sample holder could be stabilized with the accuracy ±0.2 °C in the range of 5–80 °C.

2.3. Light Transmission Measurements

To study the role of goethite nanoparticles on ferronematic LC structural changes and subsequent development of optical properties, the light transmission measurements were used. The experimental investigation was conducted in LC cells analogous, as in the case of capacitance measurements (D = 50 µm) [19]. In addition to cell covering with ITO transparent and conductive layers, the alignment layers were rubbed in a parallel direction to cell surface. The cell's glass was illuminated by linearly polarized incident green light beam in normal direction. The LC cell position provided in the case of parallel polarizers maximal transmittance was registered by photodetector. The intensity of transmitted light, after passing the LC cell, was registered by a photodetector connected with a computer, which evaluated the light transmission as a function of magnetic field or time. The complete experimental arrangement configuration has been already described [22,23]. The light transmission could be expressed as I/I_0, in the case of parallel polarizers, where I_0 is the maximal transmittance and I is the intensity of measured instant light after passing through the LC cell, respectively.

3. Results

3.1. SAW Results

Figure 1 shows the dependence of SAW attenuation on magnetic field at a linearly increasing field (rate 15 mT/min) measured for composites of E7 dopped with goethite nanorods of three different volume concentrations (10^{-3}, 5×10^{-4} and 10^{-4}), including pure E7. Magnetic field B was, in this case, oriented perpendicularly to the cell plane and by that also to wave vector $\boldsymbol{k}$ characterizing a SAW propagation. Compared to previous capacitance results obtained on composites of 6CHBT doped with goethite nanorods [20], some increase in the threshold fields of doped composites in relation to pure E7 appears to be registered, however, only the slight shift of the threshold fields for different concentrations is observed. Although the threshold field is registered for each concentration, they are not sufficiently sharp for precise determination, especially in the case when thresholds are too close. It should be also noted that for every SAW measurement, a new cell on piezoelectric substrate has to be prepared, although, this is performed with identical spacers (D = 100 µm) so that some aberrance can occur. Usually, the aberrance is less then ±5 µm. Nevertheless, the maximal value up ±15 µm can occur. More correct determination of the threshold field should be determined using results of light transmission measurements presented in the next part. The observed dependencies usually consist of three phases, which include a slight increase in the interval of 0–85 mT, followed by a more intense growth merging into saturation. The LC molecules' main process of reorientation corresponds to the more rapid increase in the SAW attenuation. Another characteristic feature is that the magnetic field influence on structural changes noticeably increases compared to pure E7, nevertheless, the increasing concentration of goethite nanorods does not automatically ensure the rise of magnetic field influence. The evident decrease in structural changes with increasing concentration is registered with SAW attenuation response measurement. The decreasing total changes of the SAW attenuation with increasing concentration could be explained by the process of some aggregate creation due to the raising concentration, which is practically lower than the total number of magnetic particles [24,25].

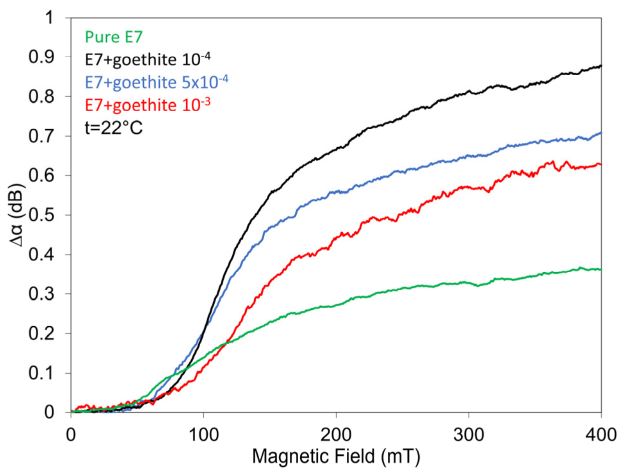


Figure 1. Dependences of SAW attenuation response on magnetic field for E7 liquid crystal doped with goethite nanorods of concentrations 10^{-4}, 5×10^{-4} and 10^{-3}, including pure E7.

Concerning the memory effect registered at a linearly decreasing magnetic field and at the same rate (15 mT/min) after reaching its maximum value (400 mT), the residual attenuation was registered at zero fields for all investigated composites (Figure 2). The values of residual attenuation increased with increasing goethite nanorods concentration, namely, from ~25% in the case of the lowest concentration (10^{-4}) up to ~60% in the case of the highest particle concentration (10^{-3}). The character of the SAW attenuation decreases was almost similar as in the increasing regime, however, for concentration 10^{-3} slight increase was registered even at beginning of decreasing magnetic field. Such behavior can consist either in the stronger anchoring of LC molecules on goethite nanorods during magnetic field application and/or in some aggregate creation. The stronger anchoring can be the reason for the process of creation of some forms that consist of nanorods and surrounding LC molecules with parallel magnetic moments. Their lifetime at the lowering magnetic field is longer than their lifetime after rapid magnetic field jump to zero (see later), as well as the lifetime of clusters formed also at decreasing magnetic fields. The aggregates could cause a process of SAW attenuation increase, which continues even in decreasing regime in the case of highest concentration (10^{-3}).

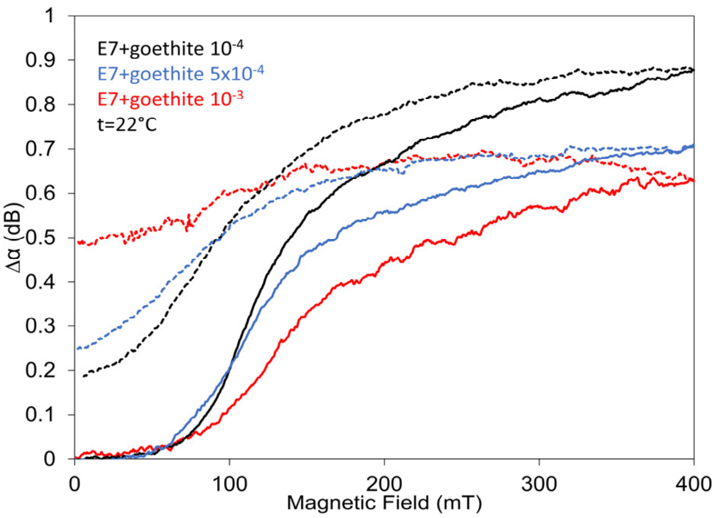


Figure 2. SAW attenuation response versus magnetic field for E7 doped with goethite nanorods of concentrations 10^{-4}, 5×10^{-4} and 10^{-3}, measured for both increasing and decreasing magnetic field.

The dependences of SAW attenuation on temperature measured for investigated LC (E7) composites consist of goethite nanorods of three different concentration (10^{-4}, 5×10^{-4} and 10^{-3}), including pure E7, which is focused on structural transitions from nematic to isotropic phase, which are illustrated in Figure 3. These dependences show for all concentrations, as well as pure LC, which has very similar progress, and it is almost at a stable value at room temperatures approximately up to the temperature of ~40 °C when the increase in SAW attenuation is registered finally up to the temperature of the nematic-isotropic transition (T_{NI}), after which a decrease in the attenuation (see also detail of Figure 3) representing structural changes to isotropic phase was detected. Presented temperature dependences show that goethite nanorods shift the nematic–isotropic transition temperature comparing to pure LC towards higher temperature. The significant shift of ~2.2 °C of the nematic-isotropic transition temperature (T_{NI}) was registered in the case of composite with the lowest concentration (10^{-4}). This trend means the additional increase in T_{NI} with increasing nanoparticles concentration continues, but in smaller steps (~0.4 °C). The reason for such behavior could be in the fact that goethite nanorods, if they have a satisfactory number of adjacent LC molecules, can produce local magnetic moments. The addition interaction can then lead to the increase in transition point [26,27]. Goethite nanoparticles behave in this case as real nanorods. The reason of difference from results observed in composites of 6CHBT liquid crystal consisting of goethite nanorods [20] can be either in different nanorods size and form or different LC properties. The interesting feature of the nematic–isotropic transition in the case of E7 is that the nematic–isotropic transition is really very slow in contrast with another liquid crystals as 6CHBT, 5CB, or 6CB [12,15,18]. The reason could be in very complicated composition of E7 that consists of four different liquid crystals. Similar results were registered also for composites E7 containing magnetosomes as host nanoparticles [18].

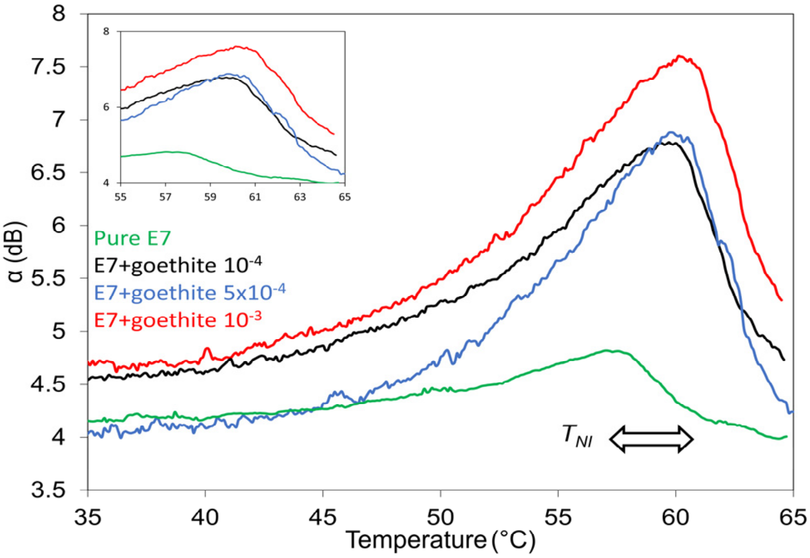


Figure 3. Dependences of SAW attenuation response on temperature for E7 liquid crystal doped with goethite nanorods of concentrations 10^{-4}, 5×10^{-4} and 10^{-3}, including pure E7.

The time development of SAW attenuation responses after application of discrete values of magnetic fields, 200 mT, 300 mT, and 400 mT to composites of E7 with goethite nanoparticles of concentration 5×10^{-4}, as representative samples presenting switching processes, are shown in Figure 4. The curve illustrating the switching process for pure E7 is on the significantly lower level, as it was registered for all investigated composites (see Figure 1), and it is not added in this figure. Very close relaxation times are characteristic for all developments when the magnetic field is removed. However, decrease in relaxation times with increasing magnetic field is registered when the magnetic field is applied. It is also evident that relaxation times of the processes occurring after the magnetic field is applied are noticeably shorter than the relaxation times of processes taking place after removing the magnetic field. The behavior, when the rate of processes occurring after the application of discrete values of the field, depends on the intensities of applied fields, coinciding with previous electro-optical [28,29] and magneto-optical [25,30] investigations. It should be noted that maximal values of SAW attenuation responses correspond quite well with the values detected in dependences of SAW attenuation on increasing magnetic field (Figure 1). The SAW attenuation responses also indicate that the concentration in investigated E7 composites play important role. The significant feature of presented time characteristics is, namely, the continual increase in SAW attenuation at 400 mT during constant magnetic field.

The reason for such behavior, as we have already mentioned, could be in the continual creation of some aggregates due to the higher intensity of magnetic field. The implication is, then, the residual SAW response after magnetic field removal.

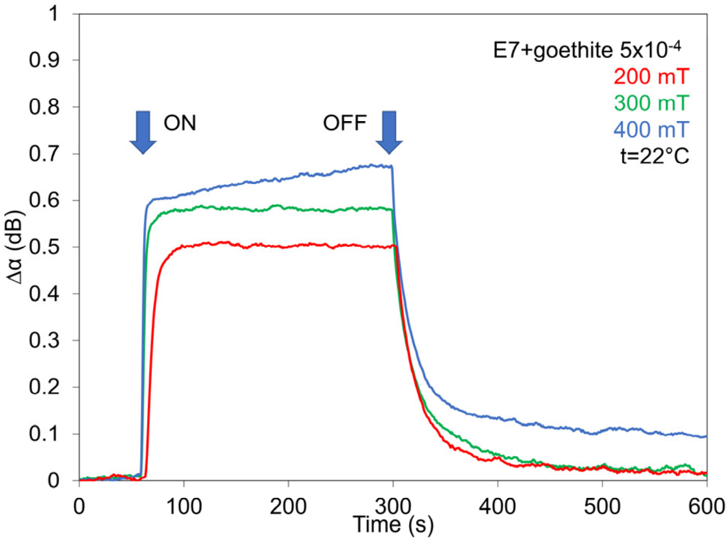


Figure 4. SAW attenuation time responses on applied discrete values of magnetic fields (200, 300, and 400 mT) for E7 doped with goethite nanorods of concentrations 5×10^{-4}.

3.2. Light Transmission Investigation

The results of the measured light transmission dependence on magnetic field for all investigated LC composites and pure E7 are presented in Figure 5. The figure illustrates the light transmission developments for increasing magnetic field B oriented perpendicularly to both cell plane and wave vector. According to pure LC, the important role of goethite nanorods on the structural changes and by that on optical properties are registered. In contrast to SAW measurements, the clear decrease in the threshold fields was registered. At first, the threshold field decreased only slightly with increasing concentration, but the decrease became more rapid at the highest concentration (10^{-3}). However, the position of the threshold field is influenced by the presence of magnetic nanoparticles, and it is due to the combination of both ferromagnetic and anchoring energies, meaning it depends on the concentration, size, and shape of the nanoparticles. The decrease in threshold values, which is required from an application point of view, is then more significant for the composites with the highest nanoparticle volume concentration. In this case, the parallel orientation of the magnetization vector and the liquid crystal director suggest that the threshold magnetic field should be set to lower values [31]. The result concerning decreasing threshold field in E7 composites with increasing concentration coincides very well with previous ones obtained in 6CHBT composites [19. The presented development of the light transmission clearly shows that, after the threshold transition, its fast decrease continues up to the minimum, followed by an increase merging into saturation. However, superimposed oscillations are registered during light transmission increase. Both the transmission minimum and oscillation amplitude are influenced by the goethite concentration so that the lowest minimum and smallest oscillation amplitudes are in the composite with highest concentration of goethite.

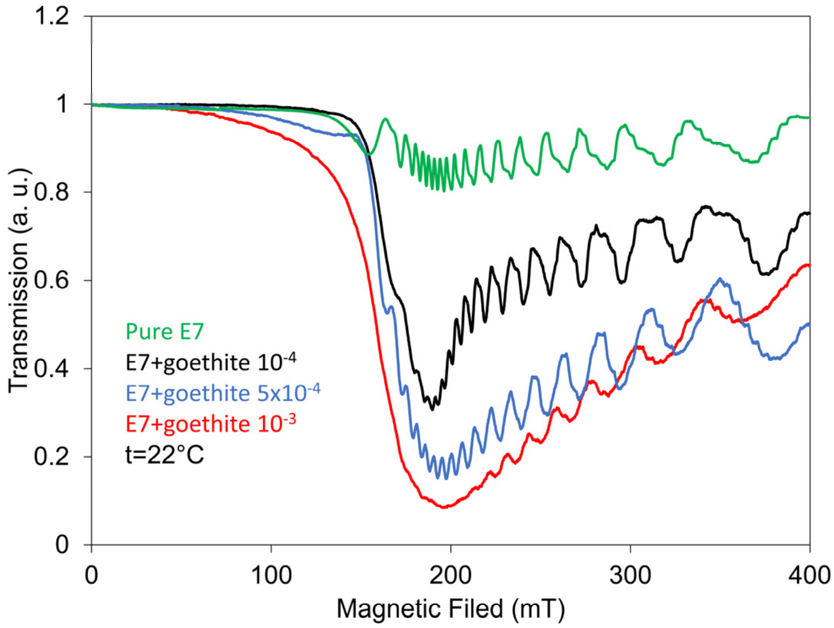
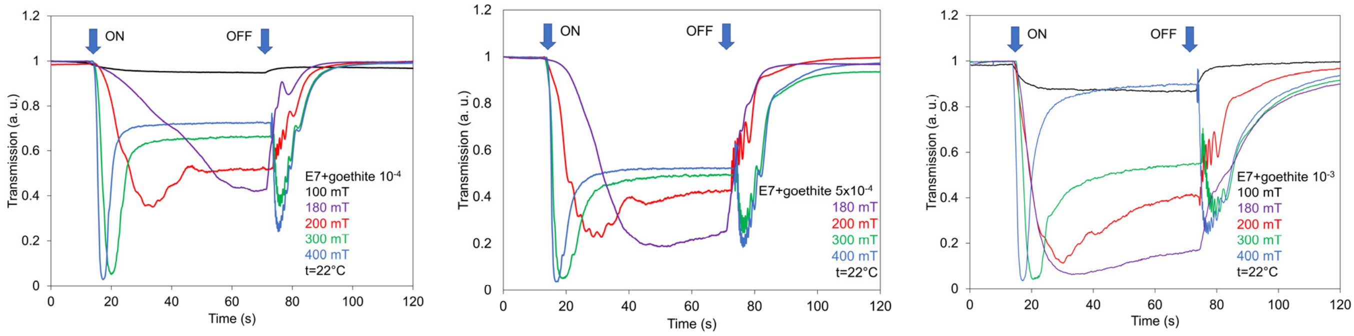


Figure 5. Dependences of light transmission on a magnetic field for E7 liquid crystal doped with goethite nanorods for the concentrations 10^{-4}, 5×10^{-4} and 10^{-3}, including pure E7.

The transmission oscillations are caused by a reorientation of the molecular director when the LC is subjected to an external field higher than the threshold field and to a laser beam. It can be explained by the directional redistribution of LC molecules around the equilibrium position of the nematic director, from initial planar alignment to perpendicular, occurring primarily at the center of the LC layer [32]. LCs transmit light through the cell in sequences of maxima and minima when illuminated with a laser beam. [23,33]. If B is turned off and the LC returns to its initial planar orientation, the same situation occurs. The order number of transmission maxima and minima, occurring when the external field is higher than the threshold field, can be a function of the maximum deviation angle, the cell thickness, and the light wavelength [34]. The oscillation shape of a saturation regime is observed at the measurements of phase difference in magnetically doped nematic LCs, which can also be understood to be a part of their collective behavior [35].

The dynamics of the switching processes for different values of jumped magnetic field (100, 180, 200, 300 and 400 mT) are shown for composites of all concentrations in Figure 6 The effect of applied field in the jumped regime with magnetic field running for 120 s corresponds to obtained magneto–optical characteristics (Figure 5). The intensity of magnetic field and goethite nanorod concentration determine the structural changes initiated by the applied field and by that corresponding light transmission development [25,28–30]. The shortening of the switching time after application of discrete value of magnetic field with increasing field intensity for all concentrations, as well as its gradual shortening with increasing concentration, can be seen. The superimposed oscillations at this part of light transmission development are negligible and, for higher magnetic fields, they even disappear. After reaching the minimum, the light transmission increases following the saturation level that corresponds to the level obtained in magneto–optical characteristics (Figure 5). There is a distinct change in light transmission when the magnetic field is removed, however, with a shallower minimum and clearer resulting oscillations. The seemingly special selection of the magnetic field value 180 mT is related to the choice of magnetic field that is higher than threshold field, but still lower than the field corresponding to the minimum of light transmission. As it can be seen, the development of light transmission is for such field characteristics by the lowest saturation level and by the most stable saturation state, without any oscillations.



(**a**) (**b**) (**c**)

Figure 6. Light transmission time responses registered on E7 liquid crystal doped with goethite nanorods of different concentration 10^{-4} (**a**), 5×10^{-4} (**b**), and 10^{-3} (**c**) after jumped magnetic field changes of 100 mT, 180 mT, 200 mT, 300 mT and 400 mT were applied.

In nematic LCs enhanced by short spherical and chain-like magnetic nanoparticles or magnetosomes, superimposed oscillations on light transmission characteristics were observed [18,22,23]. Rod-like nanoparticles, nanorods, or carbon nanotubes in LCs [12,22,23,25] exhibited different behavior after the magnetic field was applied, since light transmission and, therefore, structural changes were sufficiently stable. Such composites could be suitable for applications in switching processes. Nanoparticle shape, length, and concentration, as well as anchoring, play crucial roles in the stability of magneto–optical behavior.

4. Conclusions

In this contribution, the SAW technique and light transmission measurements were used to study the influence of goethite nanorod dopants on nematic LC (E7) behavior. Both the attenuation response of SAW and the response of light transmission on occurring structural changes confirmed the important role of goethite nanorods on the total structural changes according to pure LC. The magnetic field influence on structural changes noticeably increases compared to pure E7, however, the increasing concentration of goethite nanorods led to the decrease in structural changes registered with SAW attenuation response. The strong memory effect registered at decreased magnetic field at zero fields for all investigated composites (~25–60%), at which point the values of residual attenuation increased with increasing goethite nanorods concentration. Presented temperature dependences show that goethite nanorods shift the nematic–isotropic transition temperature compared to pure LC towards higher temperature. Goethite nanoparticles behave, in this case, as real nanorods, which is different from previous results. Light transmission experiments registered a clear decrease in the threshold field with the important feature that the threshold field shift slightly decreases with increasing concentration followed by the lowest concentration. The developments of the switching processes for various intensities of jumped magnetic field, however, showed that, due to the existing oscillations, investigated composites could be difficulty used in application as optical devices. Obtained results supported previous conclusions that the shape and length of magnetic nanoparticles included with their concentration play a critical role concerning the magneto–optical properties stability. In addition, obtained results were discussed and compared in consideration of previous results and coincide with those observed using both different magnetic nanoparticles and experimental techniques.

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