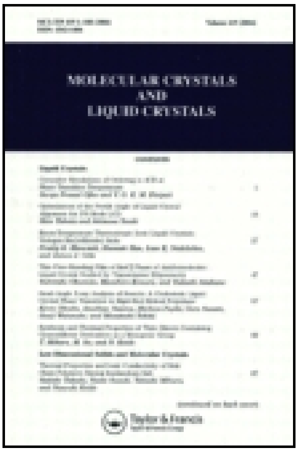

Molecular Crystals and Liquid Crystals
Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/gmcl20

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Version of record first published: 15 May 2012.

To cite this article: O. Buluy, D. Burseva, M. R. Hakobyan, J. W. Goodby, M. A. Kolosov, Yu. Reznikov, R. S. Hakobyan, K. Slyusarenko, M. F. Prodanov & V. Vashchenko (2012): Influence of Surface Treatment of Ferromagnetic Nanoparticles on Properties of Thermotropic Nematic Liquid Crystals, Molecular Crystals and Liquid Crystals, 560:1, 149-158

To link to this article: http://dx.doi.org/10.1080/15421406.2012.663195

Mol. Cryst. Liq. Cryst., Vol. 560: pp. 149–158, 2012

ISSN: 1542-1406 print/1563-5287 online
DOI: 10.1080/15421406.2012.663195

Influence of Surface Treatment of Ferromagnetic Nanoparticles on Properties of Thermotropic Nematic Liquid Crystals

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We observe a strong effect of surfactant structure on the aggregation of ferromagnetic nanoparticles in nematic liquid crystal (LC) hosts, and the colloidal stability and ordering of the resulting nematic matrix. We find that ferromagnetic nanoparticles coated with surfactants that possess terminal mesogenic groups induce minimal aggregation of the nanoparticles, resulting in a 1.5 times reduction in the value of the Frederikscz transition field in comparison with the parent LC host.

Keywords Aggregation; ferromagnetic nanoparticles; liquid crystals; surfactants

1. Introduction

Dispersions of ferromagnetic nanoparticles in thermotropic liquid crystals (LCs) are attracting intense interest by scientists and engineers because of their extreme sensitivity towards magnetic fields [1–4]. This sensitivity is due to the coupling between the dipole moments of the particles with the director field of the liquid crystal host. The origin of this interaction can be, for instance, the anisometry of the elongated particles or angularly dependent adsorption of LC molecules on the surface of the spherical particles. In both cases the reorientation of the particles towards the magnetic field results in a force that drags liquid crystals after the particles. Similar observations and property-structure correlations have been made with respect to the dielectric properties of nanoparticle dispersions in nematic liquid crystal hosts [5].

The main problem with the application of ferromagnetic LC suspensions is their stability. Although estimates show that the interaction between the nanoparticles due to

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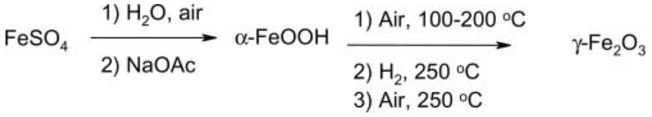
elastic LC forces are weak and the direct magnetic interactions are much weaker than the thermal energy [6], the aggregation of the ferromagnetic particles dispersed in nematic hosts is extremely common [3]. In order to suppress aggregation, nanoparticles are usually coated with various surfactants [7–10]. The main role of a surfactant is to increase the excluded volume of the particle. In other words, the surfactant systems increase the steric repulsion radius of the particles, and prevent the particles from approaching each other up to the distance of direct attractive interactions. At the same time, the presence of surfactants that have elongated molecular architectures, which penetrate into a LC matrix, can seriously alter the mesogenic characteristics of the matrix, thereby affecting the ordering, elastic and properties properties, and the phase transitions. Therefore, the problem of selection and usage of a surfactant does not necessarily totally depress aggregation, however, it minimizes the effects on the properties of LC matrices.

In this article, we report the strong effects that surfactant structure has on the aggregation of ferromagnetic nanoparticles dispersed in host nematic LC; on the stabilities of colloid systems, and on the organization within the nematic matrix. We show that surfactants which possess 4-cyanobiphenyl mesogenic units at the exterior ends of the surfactant molecules provides for minimal aggregation of the particles, and the dispersion of the particles covered with this surfactant has one and half times lower value of the Frederikscz transition field than for the comparative LC host.

2. Materials and Experiments

We studied dispersions of elongated, needle-like ferromagnetic nanoparticles γ-Fe_2O_3 covered with various surfactants in the host 4-pentyl-4′-cyanobiphenyl (5CB). These particles are promising for applications since they are ferromagnetic at ambient temperature and their synthesis is rather simple and well developed [11], and the theoretical estimations predict a strong enhancement of sensitivity of LC to magnetic field in the case of true colloid [12]. The nanoparticles were synthesized according to the following scheme [11] with the exception of intermediate α-FeOOH obtaining was adopted from work [13]:



$$FeSO_4 \xrightarrow[\text{2) NaOAc}]{\text{1) } H_2O\text{, air}} \alpha\text{-FeOOH} \xrightarrow[\substack{\text{2) } H_2\text{, 250 °C} \\ \text{3) Air, 250 °C}}]{\text{1) Air, 100-200 °C}} \gamma\text{-}Fe_2O_3$$

The average size of the particles was about 175 × 25 nm. Their TEM images are shown in Fig. 1.

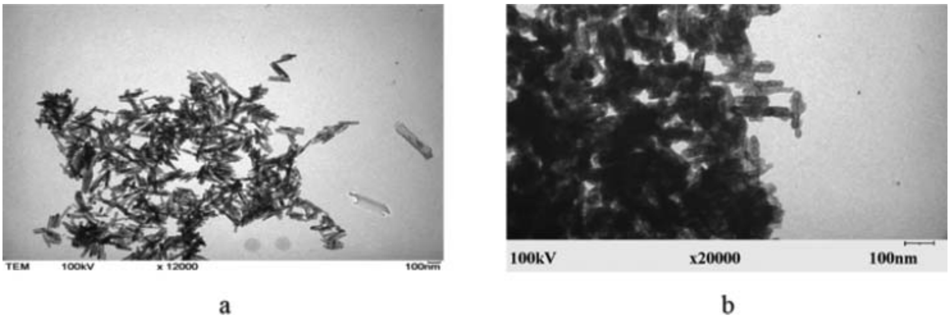
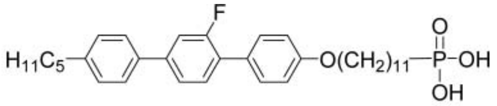
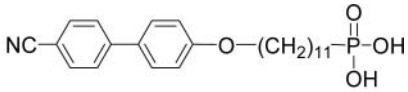


Figure 1. TEM images of intermediate α-FeOOH (a) and ferromagnetic γ-Fe_2O_3; (b) nanoparticles.

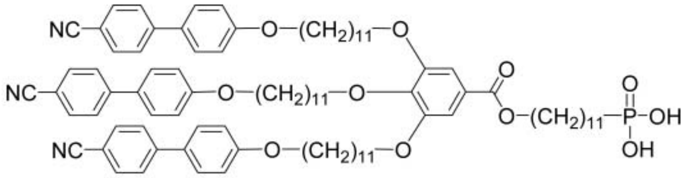
Table 1. Structure chemical formulas of the surfactants



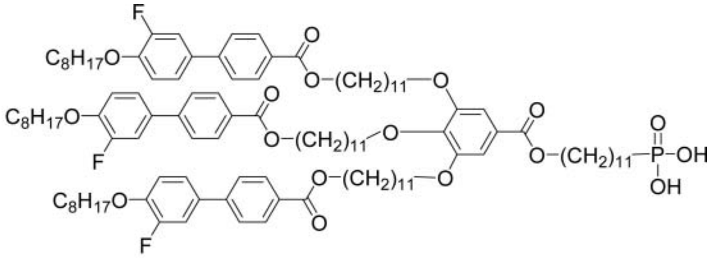
S-1



S-2



S-3



S-4

It is worth to note that although the perfect acicular shape of the intermediate α-FeOOH nanoparticles was slightly deformed upon the chemical transformation, the final γ-Fe_2O_3 nanoparticles still retained the rod-like shapes.

We studied two types of surfactants (Table 1). The first two surfactants we examined possessed linear aromatic cores (S-1, S-2), and third and fourth surfactants were dendrons (S-3, S-4). Both surfactant types possessed terminal mesogen-units. In the case of dendron surfactants, these mesogenic groups were connected to an aromatic scaffold via flexible methylene chains.

The nanoparticles were dispersed in the solution of the surfactants in toluene in the presence of small amount of pyridine over a period of 10–12 h. Afterwards the nanoparticles were washed thoroughly with hot toluene to remove any unbound surfactants and then separated by the use of a permanent magnet. The completion of the coating of the nanoparticles was monitored by FT-IR. The coated nanoparticles were then dispersed in chloroform and mixed in the LC in the appropriate proportions. The chloroform subsequently removed by evaporation under vacuum at 120°C over a period of several hours. The concentration of the particles in the LC, c_{part} was determined to be 0.05 wt.%. To vary the concentration of the particles, the dispersions were diluted until a minimal concentration, $c_{part} \approx 0.025$ wt.%

was achieved. All freshly prepared dispersions had homogeneous yellowish coloration that exhibited uniform distributions of the particles.

In order to study an influence of the coated particles on the ordering of the nematic matrix, we compared the temperatures of the phase transitions to the isotropic phase of the dispersions with the host LC. Capillaries filled with dispersions of the coated nanoparticles and the LC host 5CB were placed close to each other in the microscope hot-stage and observed via transmitted polarized light microscopy. The temperature of the capillaries was increased by 0.1 ± 0.03°C per 30 min intervals, starting from 32°C. The temperature of formation of first isotropic areas in the nematic phase, T_{c1}, and the temperature of vanishing of the last nematic areas in isotropic phase, T_{c2}, were measured for each dispersion, using the host LC as a reference. We found that the inclusion of particles changes both temperatures and the width of the two-phase region, $\Delta T_{c1-c2} = T_{c1}\text{–}T_{c2}$. The results of these measurements are presented in Table 2.

It can be seen that all of the dispersions exhibited a shift in the clearing point transition temperature and a widening of the two-phase region. Most of the dispersions reveal a decrease in the transition temperature, with the exception of the dispersion with the surfactant S-4, which demonstrates a substantial increase in the transition temperature. It is important to note that the measured values T_{c1} and T_{c2} were well reproducibal. Furthermore, the small changes of T_{c1}; T_{c2} values (tenths of degrees) can be probably explained by presence of impurities, the concentration of which varied from sample to sample. As an alternative, the origin of the large shift of T_{c1} for samples S-2 and S-4 (around 3 degrees) may be related to the surfactant-treated nanoparticles their interactions with the host.

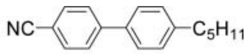
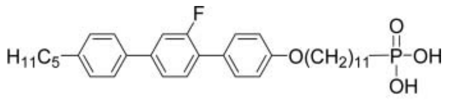
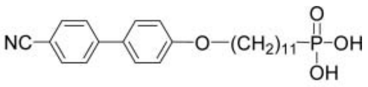
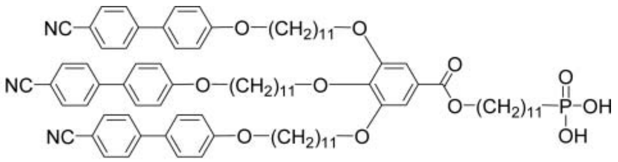
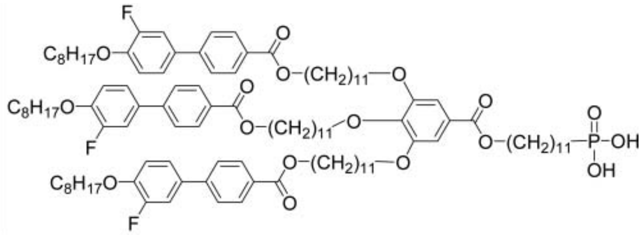
In order to estimate the stability of the dispersions, they were inserted by capillary force into the glass capillaries of length of 70 mm by 2 mm diameter at room temperature. Before the filling, the dispersions were mechanically mixed for 1 hour at 90–100°C, and then ultrasonicated for 10 minutes at the same temperature. The capillaries were mounted vertically and the optical changes in the dispersions were observed as a function of time. We observed the formation of small (2–5 μm) aggregates immediately after the filling. Their size increased with time and the aggregates gradually descended to the bottom of the capillaries. Visible formation of the aggregates stopped after 10–14 hours after filling of the capillaries for all types of the dispersions.

The rate of the aggregation and the final distribution of the aggregates over the length of the capillaries, strongly depended on the type of the surfactant used as shown in Fig. 2. Although precipitated aggregates were observed for all the samples, the sample with the particles treated with the surfactant S-2 compared favorably with respect to the others.

This sample contained no visible aggregates except at the bottom area, and it revealed the richest yellow coloring that demonstrated the presence of a high concentration of coated particles in the nematic matrix. Therefore, we focused our studies on the more promising dispersions of 5CB with surfactant S-2 in the next set of experiments we report below.

In order to follow the aggregation dynamics of the dispersions with the surfactant S-2 in more detail, we took the pictures of the dispersion in a cell thickness of $d = 50$ μm, and with planar anchoring conditions. The snapshots were made each 10 s during first 30 min. Then the snapshots were taken each minute during 1.5 hours and then each 5 min during 14 hours. The results of the digital processing of the images according to [14] allowed us to plot the dependence of the number of visible aggregates (≥ 1 μm) over time from the start of the filling (see Fig. 3). One can see the speed of aggregations formation sharply decreases at a characteristic time of about 1 hour, and the aggregation visibly stops after

Table 2. Phase transition temperatures of the dispersion of nanoparticles covered with different surfactants.

	T_{C1},^{o}	$\Delta T_{c1\text{-}2}$,^{o}
 **5CB**, (c_{part} = 0)	36,0	0,2
 **S-1**,(c_{part} = 0.05 wt%)	35,5	0,3
 **S-2**, (c_{part} = 0.05 wt%)	33,9	1,3
 **S-3**, (c_{part} = 0.01 wt%)	35,9	0,5
 **S-4**, (c_{part} = 0.05 wt%)	37,8	1,1

about 10 hours. The aggregations size fitted a normal distribution, with the maximum at about 4 μm and dispersion about of 4 μm. The aggregates possessed a slight anisometry ($\leq$1:3) and, unlike the earlier results [3], did not reveal pronounced orientational ordering.

In order to check the sensitivity of the dispersion with the surfactant S-2 to a magnetic field, we studied the magneto-optical response of dispersions of different concentrations in cells ($d = 50$ μm) with planar-aligned. The cells were placed between the poles of an electro-magnet, the field for which was controlled over a range of 0.2–2 kG. The temperature in the measured area was 25 ± 0.5°C during the studies. The director $\vec{n}$ and the magnetic field $\vec{H}$, were arranged to be perpendicular to one another.

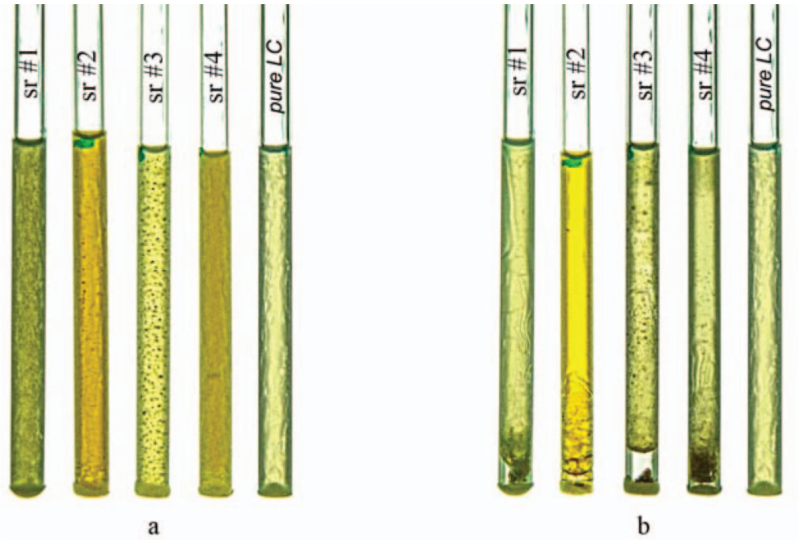


Figure 2. Photographs of the capillaries filled with the pure LC and with the dispersions of γ-Fe_2O_3 particles covered with different surfactants. a) immediately after filling; b) 19 hours after filling.

A small bias field, $\vec{H}_{bias} = 0.2$ kG was also applied along the director, $\vec{n}$, to provide initial polar ordering of the ferromagnetic nanoparticles in the LC matrix. The response of the cell to application of the field was detected by measuring the dependence of its optical phase retardation, $\Delta\Phi$, on the magnetic field $\vec{H}$. To carry out this measurement, the cell was placed between the crossed polarizers, the axis of which were arranged to be 45° with respect to director, $\vec{n}$, and the dependence of the intensity of aprobe laser beam, $I_{probe}(H)$, with respect of the magnetic field was measured. From these studies, the dependency of $\Delta\Phi(H)$ on the applied magnetic field for two samples of different concentrations of the particles (0.01 and 0.005 wt.%), c_{part}, are shown together, along with the dependency for

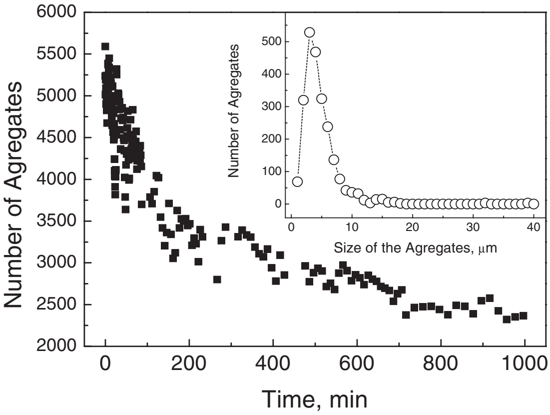


Figure 3. Dependence of the number of visible aggregates on the time since the filling of the cell. The final distribution of the aggregates by sizes is shown in the insertion.

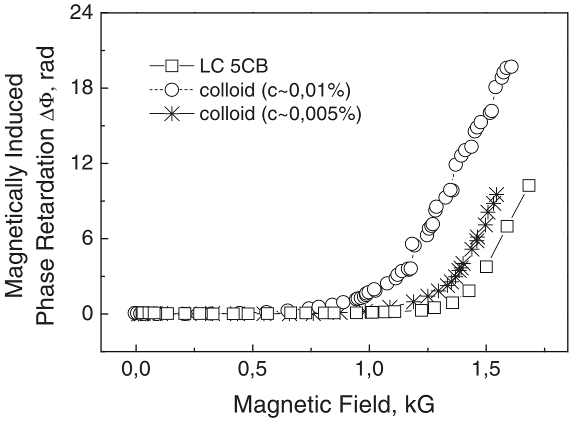


Figure 4. Dependencies of the magnetically induced birefringence on the magnetic field.

pure 5CB, in Fig. 4. The threshold-like character of the dependencies can be seen for all the three studies.

The slight spreading of the Frederikscz transition is probably caused by non-perfect planar alignment of the pure 5CB and the dispersion (the pretilt angle was about 1.5°). We determined the value of the Frederikscz transition field, H_{Fr}, as a field at which the magnetically-induced phase retardation exceeded the experimental error. It can be seen that the decrease in the value H_{Fr} with an increase in the dispersion concentration. For the dispersion with $c_{part} = 0.01$, the threshold value $H_{Fr} = 0.89$ kG is 1.5 times lower than $H_{Fr} = 1.36$ kG for pure 5CB.

3. Discussion

Our experiments showed that the ferromagnetic particles treated with the surfactant strongly affect the ordering of the LC matrix, which is manifested by the shift of the phase transition temperatures and the widening of phase coexistence temperature range of the dispersion. This changes can be caused by; i) "dilution effect" [15] due to formation in the matrix of the excluded volume not occupied by the LC; ii) the presence of a magnetic field originated from the particles; or iii) orientational interaction of the surfactant molecules with the neighboring molecules of the LC.

In the case of spherical nanoparticles, the dilution effect results in a decrease of T_c. If the particles are anisotropic, it promotes the ordering of the LC molecules and the slight increase of T_c. Both effects typically result in very slight shift of $T_c < 10^{-1}$°C [15] and cannot explain our experimental data. In order to estimate whether ferromagnetism of nanoparticles can cause notable changes of the LC ordering, the free energy of the dispersion in the volume unit in Landau—de Gennes form can be written as:

$$F\frac{1}{2}a(T - T^*)S^2 - \frac{1}{3}BS^3 + \frac{1}{4}CS^4 + F_n + F_{\text{int}} \qquad (1)$$

were S is the order parameter of a LC, T^* is the temperature of second order transition from the nematic phase to isotropic phase, a, B and C are the constants. The first three terms describe the free energy of the LC nematic matrix, the fourth term describes the free energy of nanoparticles, and the last term is responsible for the interaction of nanoparticles with the LC:

$$F_{\text{int}} = -\frac{\chi_a \bar{c}_{part}}{3} \int d^3 r\, Q^{LC}_{\alpha\beta}(r) H_{part,a}(r) H_{part,\beta}(r) \tag{2}$$

where χ_a is magnetic anisotropy, $Q^{LC}_{\alpha\beta} = S(n_\alpha n_\beta - \frac{1}{3}\delta_{\alpha\beta})$ is the tensor order parameter of LC, $n_{\alpha,\beta}$ are the axes that determine the director of the LC, $\alpha,\ \beta = x,\ y,\ z,\ \bar{c}_{part}$ is the volume fraction of the particles, thus

$$\vec{H}_{part} = \frac{3\vec{r}(\vec{r}\vec{m})}{\vec{r}^5} - \frac{\vec{m}}{\vec{r}^3}, \tag{3}$$

is a field induced by the particle, where $\vec{m}$ is a magnetic dipole moment of the particle, $\vec{r}$ is thedistance from the particle.

It is reasonable to suggest that the order parameter of a LC can be considered constant over the dispersion volume, and the main axis of the tensor $Q^{LC}_{\alpha\beta}$ (director of the LC) is parallel to the main axis of the order parameter of the magnetic dipoles of the nanoparticles $Q^{n}_{\alpha\beta} = S_n(\frac{m_\alpha m_\beta}{m^2} - \frac{1}{3}\delta_{\alpha\beta})$. In this case the expression (2) can be rewritten as

$$F_{\text{int}} = -\frac{4\pi \chi_a \bar{c}_{part} m^2}{45 r^3_{part}} S S_{part}, \tag{4}$$

where r_{part} is the radius of the particle.

Minimization of the expression (1), taking into account (4) and neglecting the other small terms leads to the following expression for the phase transition temperature of the ferromagnetic nanoparticles dispersion in a nematic LC:

$$T_c = T^* + \frac{2B^2}{9aC} + \frac{\pi \bar{c}_{part} r^3_{part}}{3C_{LC} T_{c0}} \left(\frac{32\pi^2}{675 k_B} \chi_a M^2 \right)^2, \tag{5}$$

where $M = m/v_{part}$ is the magnetic moment of the particle per unit volume, v_{part} is the volume of the particle, C_{LC} is a volume concentration of LC molecules. For typical parameters $\bar{c}_{part} = 10^{-2}$, $r_{part} = 20$ nm and for the used type of γ-Fe_2O_3 nanoparticles M_m varied from 25 to 34 emu·g^{-1} [11], $\rho_{\text{part}} = 5.3$ g·cm^{-3}, $M = M_m \rho_{\text{part}} = 201.4$ emu·cm^{-3}, $C_{LC} = 10^{21}$ cm^{-3}, $\chi_\alpha = 10^{-7}$ and $T_c = 300$ K the expected shift of $\Delta T_c = 5.3\ 10^{-17}$K turned out negligibly small.

Thus, both the dilution effect and magnetic field from the particles cannot explain the observed large shifts of the phase transition temperatures for the dispersion. Thefore, the orientational interaction of the surfactant coating molecules with respect to the neighboring molecules in the LC seems the most likely cause of the effect. The mechanism of this effect can be qualitatively explained using the Grandjean-Maier-Saupe (GMS) molecular field theory in which the value and the sign of the shift of T_c is determined by the relationship between the amplitude of the inter-molecular interaction of the LC molecules, $W_{lc\text{-}lc}$, and the amplitude of interaction of LC molecules with the molecule of the surfactant, $W_{lc\text{-}sur}$ [16,17]. If the interaction of LC molecules with the surfactant molecules is weaker than the interaction between LC molecules, the shift T_c is negative. Presumably, it is realized for the surfactants S-1–S-3. In opposite case ($W_{lc\text{-}sur} > W_{lc\text{-}lc}$) the strong orientational interaction between LC molecules and surfactant molecules increases the local ordering which results in the increase in T_c. We observed the large positive shift $\Delta T_c \approx 2^\circ$ for the sample S-4 (see Tables 1 and 2). This surfactant has well extensive dendritic structure with mesogenic group in the terminal positions. Apparently, the interaction of the mesogenic group with the LC molecules is strong and the extensive structure of the surfactant allows the LC molecules to penetrate between the surfactant branch and "smooth out" the disturbance of the order parameter of the LC around the particle, which is produced due to interaction of LC molecules with the surface of the particles. As a result, the order parameter of the LC in the surfactant layer and neighboring area becomes larger than the order parameter in the LC bulk and the clearing temperature increases.

Our results showed that although the aggregation efficiency varied, all of the surfactants investigated could not completely depress the aggregation of the ferromagnetic nanoparticles. The cause of this is a strong dipole-dipole magnetic interaction between the particles, which can be estimated by the formula

$$E_{mag} \sim \frac{m^2}{r_{p-p}^3}, \tag{6}$$

where $r_{p\text{-}p}$ is the interparticles' distance. Remanent magnetization of the used type of γ-Fe_2O_3 nanoparticles M_m varied from 25 to 34 emu·g^{-1} [11], $\rho_{\text{part}} = 5.3$ g cm^{-3}, $v_{part} = 8.6 \cdot 10^{-17}$ cm^3, and $m \approx M_m \rho_{part} v_{part} \approx 1.4 \cdot 10^{-14}$ emu. In this case $E_{mag} = k_B T$ at $r_{p\text{-}p} \approx$ 170 nm. This distance is only 6 times shorter than the average inter-particles distance, $\tilde{r}_{p-p} \approx \sqrt[3]{v_{part}\rho_{part}/(C_{part}\rho_{LC})} \approx 1\ \mu$m, where $\rho_{LC} \approx 1$ g cm^{-3}, $C_{part} = 0.05$ wt%, but much longer than the length of the surfactants tails (≈2–5 nm). Therefore, the probability for the nanoparticles to approach each other to the distance at which the attraction magnetic forces are stronger than the thermal disordering is high and the surfactant cannot completely eliminate aggregation of the particles. One can expect more stable colloids of shorter anisometric particles, lengths for which are of the order of 10 nm.

Conclusions

Our experiments clearly demonstrate that the molecular structure of the surfactant strongly affect both the orientational ordering of nematic matrix and aggregation efficiency of the nanoparticles. We find that the non-dendritic surfactant possessing 4-cyanobiphenyl moieties at the termini of the molecules provides minimal aggregation of the particles, and the dispersion of the particles covered with this surfactant demonstrates a high sensitivity of the colloid to the magnetic field. The studied surfactants can both decrease and increase the ordering and clearing temperature of the nematics matrix. In particular, the surfactant S-4 with extensive dendrite-like structure with terminal mesogenic groups leads to a large increase of the clearing temperature. We relate this effect to the strong interaction of its mesogenic group with the LC molecules of the matrix, and the extensive structure of the surfactant, which allows the LC molecules to penetrate into the forest of surfactant molecules, there by enhancing the order parameter of the LC near the particle's surface.

Acknowledgment

Financial support for this work was provided by the joint grant 5205 of National Academy of Sciences of Ukraine and Science and Technology Center of Ukraine, and to the Royal Society (International Incoming Short Visits 2008/R1).

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