Magnetic-field-induced nematic–nematic phase separation and droplet formation in colloidal goethite

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Abstract

We demonstrate the suitability of polarization microscopy to study the recently discovered (parallel) nematic–(perpendicular) nematic phase separation. This novel type of phase transition is induced by applying an external magnetic field to a nematic liquid crystal of boardlike colloidal goethite and is due to an interplay between the intrinsic magnetic properties of goethite and the collective effect of liquid crystal formation. It is shown that the intense ochre colour of goethite does not preclude the use of polarization microscopy and interference colours, and that dichroism can give valuable qualitative information on the nature of the phases, their anchoring and their sedimentation and order parameter profiles. We also apply these techniques to study ‘nematic–nematic tactoids’: nematic droplets sedimenting within a nematic medium with mutually perpendicular orientations.

Online supplementary data available from stacks.iop.org/JPhysCM/23/194108/mmedia

1. Introduction

In his long and varied career one of the major and recurrent sources of inspiration for Henk Lekkerkerker has been the seminal work of Lars Onsager on the isotropic to nematic phase transition in colloidal suspensions [1]. Onsager developed his theory of the first entropy driven phase transition based on early examples of liquid crystals found in colloidal systems [2–4]. He described the transition from the disordered isotropic (I) phase to the orientationally ordered nematic (N) phase in terms of the excluded volumes between the anisometric particles, and formulated a simple virial expansion of the free energy for dispersions of rodlike particles. The I–N phase transition can then be explained as a shifting balance between the orientational and packing entropy: at a certain concentration the gain in packing entropy will exceed the loss in orientational entropy and the system undergoes an I–N transition.

In his 1949 paper Onsager also indicated the possibilities (and limitations) to extend his theory to include charged or platelike particles and polydispersity. Starting in the 1980s Henk took up the challenge and addressed all these phenomena [5–7] but also showed completely new directions like the entropy-based smectic phase [8, 9]. Experimentally, he was the driving force to develop well-characterized anisometric colloidal model systems, rodlike boehmite [10] and platelike gibbsite [11]. For gibbsite he and his collaborators not only found the elusive isotropic–nematic

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transition and a columnar phase [7], but in recent years he also studied important applications of the role of sedimentation, interfacial phenomena and droplet formation using polarization microscopy and external magnetic fields [12, 13].

In this contribution for Henk’s 65th birthday we want to apply these concepts to goethite, a model system in between pure rods and plates, forming boardlike or lathlike particles. Aqueous goethite is stabilized at pH = 3 by its surface charge leading to somewhat larger effective particle dimensions. More concentrated goethite dispersions readily show nematic, smectic and columnar phases [14–16] and even a biaxial nematic phase [17]. The magnetic properties of these particles are very intriguing [14]. They have a permanent magnetic moment $\mu$ along their longest axis $L$, presumably because of uncompensated surface spins within their anti-ferromagnetic crystal structure. In contrast, the magnetic easy axis of the magnetic susceptibility tensor $X$ lies along the shortest particle dimension $T$. Therefore, the particles align parallel to a weak and perpendicular to a strong magnetic field.

Recently, it was found that at intermediate magnetic field strengths an initially homogenous nematic phase of goethite displays an N–N phase separation. The I–N phase transition in a magnetic field was studied before for different model systems [18–20], but an N–N phase separation was never observed. Although the phenomenon initially brought to mind the theory by Henk Lekkerkerker and one of the current authors predicting N–N separation (without field) for bidisperse systems with extremely different length ratios [21], the present mechanism is entirely different and related to the intrinsic magnetic properties of goethite.

Being widely applied as the pigment yellow ochre, goethite has a very intense colour. Inspired by the beautiful work of Henk and co-workers on colourless gibbsite [12, 13], in this study we investigate to which extent different polarization microscopy techniques can still give useful information about the liquid crystal structure during N–N phase separation. In section 3.1 we prove that, despite the strong absorption, interference colours can still be observed under certain circumstances and be qualitatively exploited. Section 3.2 shows how the use of optical compensators gives more quantitative results on the differences in refractive indices involved. Section 3.3 indicates that the dichroic properties of goethite may also serve to distinguish the two different nematic phases. Section 3.4 employs a specially designed setup with a magnetic field parallel to the optical axis of the microscope to create a greatly enhanced contrast between the two nematic phases. This is then used in section 3.5 to report the first observation of what might be called nematic–nematic tactoids: droplets of one nematic phase in a perpendicular nematic background.

2. Experimental details

2.1. Synthesis

The g29 system is the same as discussed in [22]. It was obtained by hydrolysis of iron nitrate at high pH according to Lemaire et al [23]. 800 ml of a 0.1 M Fe(NO₃)₃ solution (Acros, p.a.) was mixed with 1 M NaOH (Merck, p.a.), while stirring, until pH 11 was reached. As NaOH was added to the Fe(NO₃)₃ solution, a dark-brown precipitate formed. The mixture was then centrifuged for 30 min (Beckman Coulter Avanti J-20XP, rotor JLA-8.1000, 6000g), the supernatant was removed and the particles were redispersed in 800 ml millipore (mp) water. 1 M NaOH was added to bring the solution to pH 12 and the suspension was put into an oven, for a day, at 70 °C. The solution was taken out of the oven and the supernatant was removed. The particles were redispersed in 750 ml mp water and centrifuged at 6000g for 30 min. The centrifuging procedure was repeated twice. After the last centrifugation step, the particles were redispersed in 250 ml of 3 M HNO₃ (Merck, 65%) to electrostatically charge the particles by proton adsorption. The solution was centrifuged again at 6000g for 30 min and redispersed in 750 ml mp water. The procedure was performed three times. Following the third centrifugation step, the particles were redispersed in 250 ml mp water of pH 3 (prepared with HNO₃).

The g55 system was obtained in a similar way [24]. 1 M NaOH (Acros, reagent ACS, pellets, 97%+) was added dropwise, under stirring, to a 0.1 M Fe(NO₃)₃ (Fisher Scientific, p.a.) solution until a pH of 11–12 was reached. The precipitate was aged for 9 days after which the supernatant was removed and the sediment was washed two times with doubly distilled (dd) water and 3 M HNO₃ (Merck, p.a., 65%). After centrifuging and redispersing in dd water three times the particles were redispersed in dd water to obtain a stable dispersion in water of pH 3.

2.2. TEM

Particle size distributions were determined by transmission electron microscopy (TEM) using a Tecnai 10 and 12 (FEI company) electron microscope. The particles have a more or less rectangular boardlike shape with three different dimensions: length $L$, width $W$, and thickness $T$. The length and width of about 500 particles was measured with iTEM imaging software to determine the average length $\langle L \rangle$ and width $\langle W \rangle$ and their standard deviation $b_L$ and $b_W$. The length polydispersity is then defined as $\sigma_L = b_L/\langle L \rangle$. The thickness was difficult to determine because most particles lay on their largest-area side on the TEM grid. For each sample about 10–20 particle thicknesses were measured.

2.3. Polarization microscopy

Capillaries of 0.1 mm × 2.0 mm × 100 mm and 0.05 mm × 1.0 mm × 100 mm (VitroCom) were filled with goethite dispersions with volume fractions (φ) ranging from 7 to 13% and flame sealed at both ends. They were kept in a vertical position to establish a sedimentation equilibrium profile. I–N phase separation was observed within a day after preparation.

Phase separated samples were studied in a magnetic field using polarized light microscopy. A Bruker BE25v electromagnet with large flat poles was used to produce a homogeneous magnetic field. On one side of the magnet there was a light source and a polarizer and on the other side a microscope (head of a Zeiss Axiolab microscope) with an
Figure 1. Polarized light microscopy setup with a magnetic field in the direction of the optical axis of the microscope. The arrows show the orientation of magnetization in the segments of two stacks of the permanent magnet assembly. The resulting magnetic field $B_z$ parallel to the light beam is given by a thick arrow at the sample position. The height of the permanent magnet assembly in the $y$-direction, perpendicular to the plane of drawing, is 110 mm.

Figure 2. (a) Retardation colour spectrum as visualized through a Babinet compensator, (b) the colour spectrum corrected for the absorption by a goethite dispersion, (see also the main text) (c) idem, but at a lower concentration of goethite and (d) taken with a larger exposure time.

Table 1. Goethite particle dimensions.

<table>
<thead>
<tr>
<th>System</th>
<th>$\langle L \rangle$ (nm)</th>
<th>$\sigma_L$ (%)</th>
<th>$\langle W \rangle$ (nm)</th>
<th>$\sigma_W$ (%)</th>
<th>$\langle T \rangle$ (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>g55</td>
<td>216</td>
<td>55</td>
<td>35</td>
<td>48</td>
<td>30</td>
</tr>
<tr>
<td>g29</td>
<td>242</td>
<td>29</td>
<td>49</td>
<td>30</td>
<td>35</td>
</tr>
</tbody>
</table>

3. Results and discussion

We prepared vertical samples of the g29 system (table 1) showing I–N phase separation, focusing our studies on the nematic phase. Polarization microscopy was used to study its behaviour in a magnetic field. It was found that phase separation takes place within the nematic phase when reaching a certain critical magnetic field [22]. This is around the field where particles rotate from parallel to perpendicular to the field. The two nematic phases formed have orthogonal orientations, one parallel and one perpendicular to the field. The origin of this phase separation was attributed to these two extreme types of particles that experience a maximum excluded volume and will therefore gain a lot of packing entropy by phase separating into two nematic phases with different orientations. The boardlike shape of goethite particles makes the situation slightly more complicated [22]: the upper, parallel nematic phase is uniaxial ($N^+$) and the lower perpendicular one biaxial ($N^-$), as the magnetic easy axis lies along the thickness $T$ of the particles.

3.1. Birefringence: interference colours

A closer look at the different nematic phases was taken by studying their birefringence. The birefringence $\Delta n$ of the nematic phase gives rise to a retardation or optical path difference (OPD) of the polarized light that travels a distance $t$ through the sample [25]:

\[
\text{OPD} = \Delta nt. \tag{1}
\]

This results in a sequence of retardation or interference colours with increasing retardation, as visualized with a Babinet compensator in figure 2(a). Due to strong absorption by the yellow ochre goethite dispersions, this colour spectrum changes drastically. We obtained a corrected colour spectrum...
by placing an isotropic goethite dispersion behind the Babinet compensator, as shown in figures 2(b)–(d). We find that the blue and purple tints are absent in the corrected spectrum, and also that the observed colours depend considerably on the camera settings, i.e., the exposure time.

Without doing quantitative measurements, the interference colours observed with polarization microscopy can already give valuable information about the birefringence. Bright interference colours indicate a small birefringence, a decreasing intensity of the colour bands indicates an increasing birefringence as can be seen in figure 2.

Within a uniaxial nematic phase the birefringence ($\Delta n$) increases with the volume fraction ($\phi$) and the nematic order parameter ($S_2$) according to

$$\Delta n = \Delta n_{sat}\phi S_2\tag{2}$$

where $\Delta n_{sat}$ is the specific birefringence (the birefringence limit when $\phi$ and $S_2$ are both 1) [23]. The specific birefringence is a combination of intrinsic birefringence due to the anisotropy of the crystal lattice and so-called form birefringence that is a result of the anisometric particle shape [23].

In an N–N phase separated sample, the lower, biaxial nematic phase ($N_b^-$) has an orientation perpendicular to the field. In the configuration used here, the field was applied along the width of the capillaries (figure 3). Therefore, the particles that are aligned perpendicular to the field can be aligned not only with their long axis along the wall (a), but also perpendicular to the wall ((b), homeotropic) and all orientations in between. This is different from the upper nematic phase ($N^+$), which is aligned along the field, and therefore the particles will always have their long axis along the capillary wall.

In equation (2), $S_2$ can be used for the parallel, uniaxial nematic phase because the two smaller dimensions of the particles have no preferred orientation. For the perpendicular, biaxial nematic phase the situation is more complicated and $S_2$ is not the correct order parameter to use. Furthermore, there are two different $\Delta n$s involved here. The extreme cases that might occur in the situation of alignment perpendicular to the field can be seen in figure 3. If the particles are aligned along the wall of the capillary (a) the difference in refraction along the length and thickness is important and a different order parameter should be used here. However, around the critical field strength, where the measurements in this article are performed, the biaxial order is still small and the situation is probably close to the uniaxial nematic phase.

The particles can also be homeotropically aligned (figure 3(b)), then the orientation of the width and thickness is important. Clearly, another order parameter and $\Delta n_{sat}$ will be important here. If a complete alignment along the long axis is assumed, the birefringence can be given as

$$\Delta n = \Delta n_{sat}\phi\cos 2\psi\tag{3}$$

with $\psi$ the azimuthal angle describing the particle orientation in the plane perpendicular to the nematic director. In this case, $\Delta n_{sat}$ is defined by the index of refraction along the width and thickness of the particles, while for the uniaxial case (equation (2)) $\Delta n_{sat}$ is determined by the difference between the refractive index along the long axis and the average in the other two directions.

If the order is high, a significant birefringence is expected for the situation in figure 3(b). However, around the critical field strength the biaxiality is not well developed yet and there will be a low order causing a small birefringence. This has been observed before using homeotropic samples for birefringence measurements in a magnetic field [26]. A small negative birefringence ($-0.002$) was observed around the critical field strength and it was increased by increasing the field. In a field of 1 T it was still lower than that of a uniaxial nematic phase which was aligned parallel to a small field ($-0.02$ compared to 0.06).

An example of the lower part of an N–N phase separated sample, in a field of 250 mT, can be seen in figure 4. The upper, parallel, nematic phase did not show interference colours; it had a good alignment along the field. The lower, perpendicular and biaxial, nematic phase showed intense interference colours, indicating a small birefringence. After switching off the field, this part of the nematic phase was observed as a dark region. This suggests that the particles had a homeotropic alignment, which indeed gives a smaller birefringence than alignment along the wall. The sequence of interference colours within the field betrays a gradient in concentration and order parameter (cf equation (3)) and is due to a sedimentation profile [12, 27].

In another sample, measured in a field of 200 mT, different regions were observed within the lower, perpendicular nematic phase (figure 5). In region a, again interference colours were observed; this region became partly black after the field...
Figure 4. Polarization microscopy pictures of an N–N phase separated sample (g29, $\phi = 9\%$) in a field of 250 mT and immediately after switching off the field.

Figure 5. Polarization microscopy pictures of an N–N phase separated sample (g29, $\phi = 9\%$) in a field of 200 mT and immediately after switching off the field.

was switched off and it partly showed some yellow colour. This region apparently has particle orientations perpendicular to the wall or at least close to that. Regions b did not show interference colours and seemed to be similar to the upper nematic phase. However, they are presumably different since there is a much more gradual variation of the particle orientation across the interface between regions b and a than between region a (or b) and the upper nematic phase, which is characterized by a sharp dark line. So, region b probably had an orientation perpendicular to the field, but with the long axis along the capillary wall. Region c was still a droplet of the parallel nematic phase as could be seen from the more pronounced interface around this region.

It was expected that the capillary walls would have an influence on the particle orientation, so that the particles would be aligned with their long axis along the wall. Apparently, the influence is not that large because large regions with other orientations were observed.

3.2. Birefringence: optical compensator

For a more detailed analysis of the birefringence, a 50 $\mu$m thick capillary with an I–N phase separated sample was studied in a field of about 270 mT (perpendicular to the optical axis). N–N phase separation occurred and the sample was left standing over night (figure 6). The parallel and perpendicular orientations of the different regions were confirmed using an optical compensator. In this case, the particles mostly had an orientation parallel to the wall. Within the perpendicular area (A) there were some regions (e.g. inside the solid ellipses) that showed interference colours (not very clear in the picture), indicating a smaller birefringence. These are areas where the particle orientation was tilted compared to the orientation along the wall.

Within the parallel area (B) some regions were also observed that showed interference colours and had a smaller
Figure 7. Polarization microscopy pictures of an N–N phase separated sample with different orientations of the polarizers.

Figure 8. Nematic–nematic phase separation observed with the magnetic field along the optic axis of the polarization microscope, g55 system, $\phi = 10\%$. The width of the capillary is 2 mm.

birefringence (e.g. inside the dashed ellipse). So, here there were also some regions tilted away from the perfect alignment. This was not expected because, in this configuration, particles parallel to the field would always be parallel to the wall as well. It seems that if a sedimenting droplet with the perpendicular orientation passes through a parallel area it leaves a flow-induced trace where the particles are tilted away from the ideal alignment. It was seen that this area was slowly becoming better aligned in time and finally became homogeneous.

The birefringence was measured in areas A and B and the obtained values of $-0.08$ and $0.1$ respectively were a bit higher than the measurements of Lemaire et al, who found a value of 0.064 for the aligned nematic phase [23]. This can partly be ascribed to the slightly higher volume fraction of the nematic phase in our sample (about 11% compared to 8.5%). Furthermore, it has to be mentioned that the birefringence values are subject to large errors because of the thickness of the capillaries; $50 \mu m$ seems still to be too thick. Important factors playing a role here are absorption, scattering and dichroism. The values measured seemed to be overestimations of the real values. In the solid and dashed ellipses the measured birefringence was $-0.03$ and $0.03$ respectively, which is clearly smaller than that of the more homogeneous part of these regions. This indicates an orientation away from parallel to the wall in the solid ellipses and a distortion of the alignment in the dashed ellipse.

3.3. Dichroism

The same sample was also studied with different orientations of the polarizers. With the polarizer and analyzer vertical and horizontal respectively, the full capillary became dark as is expected if the particles are aligned in the vertical and horizontal direction (figure 7(a)). The interface between different regions and the area with more defects were still visible, because there were some intermediate orientations present. Furthermore, it was noticed that the contrast with parallel polarizers was actually very good. The parallel region became bright with vertical polarizers (figure 7(b)) and the perpendicular region with horizontal polarizers (figure 7(c)). This indicates a clear difference in absorption along the different particle axes, i.e. dichroism, with the strongest absorption for light polarized parallel to the long particle axis.

3.4. Improving contrast: a magnetic field along the optical axis of the microscope

In order to distinguish the two different nematic phases more easily a new polarized light microscopy setup was developed, which is explained in detail in section 2.3. A variable permanent magnet was constructed with the field parallel to the optical axis of the microscope. Then, if the particles in the nematic phase are aligned parallel to the field and thus parallel to the optical axis, the phase will not be birefringent. However, when the particles in the nematic phase orient perpendicular to the field and the optical axis, the phase will become birefringent.

An example of nematic–nematic phase separation observed with this new setup can be seen in figure 8 and the movie in the supplementary material (available at stacks.iop.org/JPhysCM/23/194108/mmedia). The two nematic phases are indeed easy to distinguish, one is dark and the other one is birefringent.

3.5. Application to droplets

Now, it is possible to study some aspects of the phase separation process in more detail. Here, we will focus on the droplets of perpendicular nematic phase in a background of parallel nematic phase.
We observe that small droplets do not sediment, the droplets need a diameter of about 40 μm to be heavy enough to start sedimenting. The larger sedimenting droplets coalesce with the smaller droplets they find on their way while going down in the capillary (see also the movie in the supplementary material available at stacks.iop.org/JPhysCM/23/194108/mmedia).

A sample of system g29 with an overall volume fraction of 10% was studied in more detail, in a magnetic field with a strength of 250 mT. Figure 9 shows droplets with a particle orientation perpendicular to the field, captured after 22 h in the field. With the polarizers in diagonal orientation the droplets have a large birefringence, while most of the droplets are clearly darker with the polarizers in horizontal–vertical orientation. This suggests that the particles inside the droplets mostly have a horizontal or vertical orientation. Furthermore, with the polarizers in horizontal–vertical orientation the interface of the droplets displays bright birefringence with darker regions at the top and bottom as well as at the sides. The dark regions at the top and bottom are significantly narrower than at the sides. This suggests a director field in the droplets like that shown in figure 10, implying that the particles prefer planar anchoring to the nematic–nematic interface. The concentric rings visible with the polarizers in diagonal orientation indicate a decreasing birefringence when going from the drop centre to the edge, presumably caused by a decrease of the optical path length through the drop.

More precise information about the orientation of the particles inside the droplets can be obtained by using a retardation plate, a birefringent filter that adds a fixed retardation to all the wavefronts in the optical path. Depending on the respective orientation of the slow axes of the nematic phase and the retardation plate, this results in an addition (parallel orientation) or subtraction (perpendicular orientation) of the retardation of the plate and the nematic phase. Here, we use a λ/4 plate with λ = 546 nm, giving a retardation of 136 nm.

The drop in figure 11 was imaged after five days in the field. Without a retardation plate (figure 11(a)) we observe in the drop centre an orange retardation colour corresponding to a retardation of about 1450 nm (see the colour spectrum in figure 2). Insertion of the retardation plate with the slow axis oriented horizontally (figure 11(b)) leads to a colour shift to green, which corresponds to a retardation of about 1300 nm. This is a subtraction of the retardation, implying that the particles indeed have a vertical orientation. Likewise, rotation of the retardation plate to a vertical orientation (figure 11(c)) leads to a colour shift to red in the droplet centre, corresponding to a retardation of about 1600 nm, which is an addition of the retardation and confirms again the vertical alignment of the particles inside the droplet.

Interestingly, small droplets are more or less spherical, while the droplets become increasingly elongated with increasing size. Figure 12 shows relatively large droplets with a highly elongated shape between parallel polarizers. With the light polarized perpendicular to the long droplet axis the droplets appear considerably brighter than in the parallel case. Using the result obtained in section 3.3 for the dichroic properties of goethite, we deduce that the particles inside these elongated drops also have a vertical orientation. The observed elongation with increasing droplet size is in sharp contrast with nematic tactoids in an isotropic background in suspensions of rods or plates, which become more and more spherical for increasing droplet size [28–30].
A droplet of perpendicular nematic phase in the parallel nematic phase, without (a) and with ((b) and (c)) retardation plate (red arrow). The width of the images is 0.10 mm.

A strong indication that gravity plays an important role in the observed elongated droplet shape is shown in figure 13, where we observe a sequence of retardation colours in the droplet that is highly asymmetric. The largest birefringence is found at the bottom part of the droplet, implying that there is a density gradient (maybe combined with a gradient in the order parameter) inside the droplet with the highest density at the bottom.

### 4. Conclusion

The recently discovered magnetic-field-induced (parallel) nematic–(perpendicular) nematic phase separation in goethite [22, 31] can be studied using polarization microscopy, despite the intense colour of goethite dispersions. Interference colours due to sufficiently small optical path length differences can be distinguished and be interpreted using a modified birefringence colour spectrum, although care should be taken since actual
colours depend on goethite concentrations, camera sensitivity and exposure times.

In configurations with the magnetic field perpendicular to the optical axis, the uniaxial nematic phase aligning parallel to the field does not show variations in colour due to its high order parameter. The phase with its main director perpendicular to the field only displays different colours when it is also oriented perpendicular to the walls of the sample, which is due to its relatively weak (induced) biaxiality within the magnetic field. In the case that the main director is perpendicular to the field but remains parallel to the walls, no colour differences are observed either so that only more quantitative measurements of birefringence using optical compensators can give more definite answers. Quantitative values of the birefringence determined are in reasonable agreement with those previously found for single nematic phases [23]. The dichroic nature of goethite particles can also be used to distinguish different orientations.

A newly developed magnetic setup enabled us to apply a magnetic field along the optical axis. In this case the parallel uniaxial nematic phase is completely extinguished between crossed polarizers and the contrast with the perpendicular nematic phase is much clearer. We used this setup in combination with a retardation plate to study the form of and director patterns within a novel type of ‘tactoids’: droplets of the perpendicular nematic phase within a parallel nematic medium. The goethite particles are found to be anchored preferentially with their long axis parallel to the interface. Furthermore, the droplets become increasingly elongated with increasing drop size, which might be attributed to gravitational forces.

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