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Rasmita Sahoo & Surajit Dhara

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Rheological studies on liquid-crystal colloids prepared by dispersing spherical microparticles with homeotropic surface anchoring

Rasmita Sahoo and Surajit Dhara

School of Physics, University of Hyderabad, Hyderabad, India

ABSTRACT

We report rheological studies on the liquid-crystal colloids prepared by dispersing silica microparticles with homeotropic surface anchoring in 8CB liquid crystal. In nematic colloids, a shearthickening behaviour is observed in low shear rate region. The apparent yield stress of both the nematic and smectic-A (SmA) phases increases with increasing volume fraction of particles (ϕ). The critical strain amplitude (γ_c , i.e., crossover of G' and G'') in SmA colloids decreases significantly with increasing ϕ . The frequency-dependent storage modulus of SmA colloids show a power-law behaviour ($G'(\omega) \sim \omega^{\alpha}$) and the loss modulus ($G''(\omega)$) exhibits a shallow minimum. The optical rheomicroscopy shows that the nematic colloids form a network structure which are stretched and broken at high shear rate. When the temperature is decreased to SmA phase, the networks collapse showing regions of high-density particles. The variation of storage modulus with ϕ indicates that the SmA colloids response is dominated by defects.



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1. Introduction

The colloidal dispersion in isotropic liquids like water, oil etc. has been studied since decades due to their wide range of applications. However, the attention towards the dispersion of nano- and microparticles in anisotropic liquids like lyotropic and thermotropic liquid crystals (LCs) has created an immense interest in last two decades [1–6]. These are known as LC colloids. Usually, the nanoparticles do not create any longrange elastic distortion in the nematic liquid crystal (NLC) but the physical properties of the composites become significantly different from that of the pure LCs and such materials are potential for several applications [7–9]. When microparticles are dispersed in nematic LCs, they induce topological defects and create elastic distortions. This results long-range forces among the particles in the medium. These forces have no analogue in common colloidal systems where particles are dispersed in the isotropic liquids. Usually, three different types of topological defects are induced by spherical microparticles in NLCs, namely, hyperbolic hedgehog, Saturn ring and boojum depending on the anchoring strength, the elastic properties of the LCs and the particle size [1,4,10,11]. There have been many interesting experimental studies reported on LC colloids. In most studies, the attention has

CONTACT Surajit Dhara Sdsp@uohyd.ernet.in School of Physics, University of Hyderabad, Hyderabad 560046, India Supplemental data for this article can be accessed here.

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been paid on the topological defects induced by a variety of particles or the laser assisted self-assembly, manipulation of defects by optical tweezers [3,4,12–14].

However, a few studies have been reported on the bulk and rheological properties of LC colloids. For example, Oswald et al. studied the rheology of a lyotropic lamellar phase, where they showed under shear flow how the dislocation loops cross and connect the layers and disturb the flow [15]. Meeker et al. have found an unusual soft-solid state in 5CB (pentyl cyanobiphenyl) LC colloids prepared by dispersing PMMA (polymethylmethacrylate) nanoparticles (250 nm) [16]. Petrov et al. showed that solid cellular structures are formed in these systems [17]. Anderson et al. studied the details of the morphology of the cellular microstructures [18]. Subsequently, they studied the mechanical properties of the nematic matrix with the cellular morphology [19]. The origin of cellular structure was studied and discussed by Vollmer et al. [20,21]. Raghunathan et al. studied the elastic properties (splay and bend) of lyotropic LC colloids and found no substantial change in the elastic constants in the low particle concentrations (<2%) [22]. Poulin et al. have studied the phase separation and morphology of lyotropic colloidal systems made of small particles (60-120 nm latex polyballs) [23]. Zapotocky et al. investigated the effect of bigger silica particles (about 1 µm) in cholesteric LCs. From the rheological studies, they showed the effect of network of disclinations connecting the bigger clusters of particles [24]. Wood et al. dispersed nearly a micron-sized particle in 5CB and showed that the elasticity of the LC colloids increases beyond a certain concentration. They showed that it was due to the formation of percolating network structure of disclination lines. These lines are entangled with the particles giving a self-quenched defect glass state of line defects [25].

Bandyopadhyay et al. studied the rheology of LCaerosil (7 nm) composites of smectic-A (SmA) LC (8CB) and reported a soft glassy behaviour of the composites with increasing particle density [26]. Recently, Kulkarni et al. have reported on the effect of particle loading, particle shape on the rheology and the phase-transition kinetics of lyotropic hexagonal LCs [27]. In all the above experiments, topological defects induced by the particles play a crucial role and these defects were induced mostly due to the spontaneous anchoring of the molecules on the particle's surface. In the present study, we investigate the rheological properties of LC colloids prepared by dispersing silica microparticles in 8CB LC. The particles are treated for homeotropic alignment of the LC molecules at the surface that usually induce hyperbolic hedgehog, Saturn ring or escaped defects (bubblegum defects) depending on the surface anchoring and confinement. We performed rheomicroscopy studies that reveal shear-dependent microstructural change of the nematic colloid that gives shear thickening. Our study suggests that with increasing volume fraction, the SmA colloids tend to emerge from soft glass to shear induced ordered state of colloids.

2 Experimental

In the experiment, we used monodisperse silica microspheres (average diameter 5 µm), coated with octade-(3-trimethoxysilylpropyl) cyldimethyl ammonium chloride (DMOAP) to obtain perpendicular (homeotropic) alignment of LC molecules on the surface. Relatively larger size of the particle was chosen (than previously reported) for the purpose of simultaneous optical rheomicroscopy studies. The microspheres with different volume fractions (ϕ) namely 1–20% were dispersed in the nematic phase of 8CB LC by using a vortex mixture and ultrasonication. Rheological measurements were made by using a controlled strain rheometer (Anton Paar MCR-501). The optical rheomicroscopy setup consists of a CCD colour camera (model: Lumenera), a microscope tube and a long working-distance objective (NA = 0.4, $20\times$, Nikon) [28]. A polariser and an analyser were kept crossed and placed in the tube in appropriate places. We used parallel plate geometry of diameter 43 mm provided by Anton Paar for rheomiscroscopy studies. The thickness of the bottom glass plate is 6 mm. The sample was illuminated by light source through the objective from the bottom and the images were taken in the reflection mode. The microscope tube is adjustable in both the horizontal and vertical directions for the focusing purpose. The gap maintained between two parallel glass plates was 0.075 mm. The temperature of the sample was controlled by a Peltier temperature controller. The compound 4-cyano-4'octylbiphenyl (8CB) was obtained from Sigma-Aldrich and exhibits the following phase sequence: I 39.7°C N 32.4°C SmA. These temperatures are measured by the Peltier temperature controller of the rheometer. All the experiments were performed in cooling the sample from the nematic to SmA phase.

3. Results and discussion

3.1. Nematic colloids

First, we observed the textures and the phase-transition temperatures of sample using polarising optical



Figure 1. Rheomicroscopy image taken under crossed without shear for $\phi = 1\%$ in the nematic phase (35°C). Four bright lobes showing the director distortion due to the Saturn ring defects associated with the particles.



Figure 2. (a) Shear rate-dependent shear stress (σ) and (b) yield stress (σ_y) at various volume fractions at a fixed temperature ($T = 35^{\circ}$ C).

microscope. There was no appreciable change in the nematic-isotropic and nematic-SmA phase-transition temperatures. This was further confirmed by differential scanning calorimetry studies (see Supplementary Information) [29]. The sample was mounted on the rheometer's plate in the nematic phase.

Figure 1 shows the texture observed under rheomicroscope after mounting (without shear). Most of the particles are agglomerated in smaller domains and only a few of them are dispersed individually. The isolated particles in Figure 1 show four visible-coloured lobes of director distortion suggesting they have quadrupolar director configuration [25].

The sample was presheared at a low shear rate $(\dot{y} = 10 \, \text{s}^{-1})$ for 5 min before starting the measurements. Figure 2(a) shows the shear rate-dependent shear stress at different concentrations of particles in the nematic phase (35°C). The apparent yield stress (σ_{ν}) is increasing with increasing volume fraction of particles. Above shear rate 10 s⁻¹, the stress is proportional to the shear rate and this behaviour is typical for nematics at high shear rate. Figure 2(b) shows the variation of yield stress (σ_{ν}) as a function of volume fraction (ϕ). Up to $\phi = 5\%$, it shows a small but linear increase and beyond this σ_y changes slope and increases rapidly. For example, σ_v at $\phi = 20\%$ is approximately 200 times larger than that of the pure 8CB (Figure 2(b)). The linear increase of stress is expected in dilute colloidal systems (Einstein's relation). The rapid increase in the σ_v suggests that there are some collective response of the particles.

To get more insight into the dilute systems, we looked at the textures in the rheomicroscope and measured the shear rate dependent viscosity at various temperatures. The sample was presheared at a high shear rate ($\dot{\gamma} = 500 \text{ s}^{-1}$) for 5 min to disperse the particles.

Figure 3 shows the variation of apparent viscosity at $\phi = 1\%$ in the nematic phase at various temperatures. A clear shear-thickening behaviour is observed in the nematic phase (with particles) at all temperatures in the low shear rate range. This behaviour is significantly different than the shear-thinning behaviour of nematic without particles (open circles in Figure 3).

Figure 4 shows some representative images taken at different shear rates at temperature 33.5°C for $\phi = 1\%$. With increasing shear rate, different microstructures of the particles are observed. In the low shear rate range $(0.01-0.4 \text{ s}^{-1})$, the colloidal chains are bridged to form a network structure. The increase of the yield stress (Figure 2(b)) with volume fraction of particles is due to these network structures. In the shear-thickening region, the network is stretched, accompanied by deformation of the bridging chains. Increasing the shear rate increases the stress on the network structures and once the critical force is exceeded (about $\dot{\gamma} = 0.6 \, \text{s}^{-1}$), they begin to break into smaller chains showing a shear-thinning behaviour. The shear thinning continues as long as the breaking process continues. At higher shear rate ($\dot{\gamma} = 39.8 \, \text{s}^{-1}$), the chains start to break and forms relatively shorter chains. This shear-thickening mechanism is somewhat similar to those reported in micelle forming associating polymers in water [30]. At much higher shear rate $\dot{\gamma} = 100 \, \text{s}^{-1}$, many chains tend to align normal to the shear direction. At the same time, many long disinclination lines are appeared (Figure 4, $\dot{\gamma} = 100 - 150 \,\text{s}^{-1}$). These line defects could be similar to those observed in microchannels by Sengupta et al.[31]. With increasing shear, the nematic director is orientated more uniformly and the nearly perpendicular orientation of the chains is consistent with the quadrupolar defect structure. In particular, in aligned cells, the quadrupolar chains



Figure 3. (Colour online) Variation of apparent viscosity η at a few temperatures at concentration $\phi = 1\%$.



Figure 4. (Colour online) Representative rheomicroscopy images taken during the measurement of shear rate-dependent shear viscosity in the nematic phase at 33.5°C for $\phi = 1\%$. Red arrows indicate the disclination lines.

tend to align perpendicular to the nematic director. At very high shear rate (above $\dot{\gamma} = 200 \,\mathrm{s}^{-1}$), the short chains further break into individual particles which are connected by the disclination lines. This can be understood based on the competing effect of shear energy and elastic interaction energy of the particles. The approximate shear energy of a particle can be written as $E = 6\pi\eta \dot{\gamma}ha^2$, where η is the apparent viscosity, h is the gap between the plates and a is the radius of the particles. The estimated shear energy at $\dot{\gamma} =$ $20 \,\mathrm{s}^{-1}$ is about $10^6 k_B T$. Typical elastic interaction energy per particle is about a few thousand $k_B T$ [4]. However, this energy is expected to be a few orders of magnitude larger in the network (because a large number of particles are involved) and comparable to the shear energy. When the shear energy exceeds the elastic interaction energy, the breaking of chains into individual particles is expected.

3.2. SmA colloids

Induced defects and colloidal interaction in SmA LCs are not well understood so far. There are a few studies on the transformation of the topological defects across the nematic–SmA phase transition [12–14] in uniformly aligned cells. The induced disorder by the foreign particles in the SmA phase is expected to affect the mechanical properties. Hence, we carried out



Figure 5. (Colour online) Some representative rheomicroscopy images taken across the N–SmA phase transition (shear rate $\dot{\gamma} = 1 \text{ s}^{-1}$).

rheological measurements in the SmA phase. Figure 5 shows some representative rheomicroscopy images taken across the N–SmA phase transition. We observed that as the SmA phase is approached, the networks tend to get compressed and in the SmA phase, the particles are collapsed creating focal conic texture. The regions without particles are well aligned (no focal conic texture). Unlike the nematic phase, no colloidal chains are observed in the SmA phase. Isolated colloidal particles with quadrupolar defect across the N–SmA phase transition have been studied by us in planar-aligned cell [14]. The Saturn ring disappears at the transition and there is a boundary condition violation at the interface, and clear layer structure around the particles cannot be directly deduced from the optical micrographs [14].

Figure 6(a) shows the variation of shear stress in the SmA phase (28°C) at various volume fractions. The apparent yield stress (σ_y) increases with increasing volume fraction of particles. For example, σ_y for pure SmA is about 0.33 Pa and it increases to 51 Pa at $\phi = 20\%$; hence, this is about 150 times larger (Figure 6(b)).

Figure 7 shows the variation of storage (G') and loss (G'') moduli, as a function of applied strain γ at different volume fractions. Two important observations are made



Figure 6. (Colour online) (a) Shear rate-dependent shear stress in the SmA phase (28°C) at various volume fractions of particles. (b) Variation of yield stress (σ_v) with ϕ obtained from Figure 6(a).



Figure 7. (Colour online) (a)–(e) Strain dependence of the storage G' and loss G'' moduli with increasing volume fraction of particle at constant angular frequency $\omega = 1 \text{ rad/s}$ at $T = 25^{\circ}$ C. The arrows indicate the crossover. (f) The variation of critical strain amplitude, γ_c (the crossover of G' and G''), at different volume fractions.

[1]; both the elastic moduli (G' and G'') increase and [2] the critical strain amplitude γ_c (crossover of G' and G'') decreases with increasing ϕ . For example, G' is 300 times larger in LC composite with $\phi = 20\%$ compared to the pure 8CB. The variation of γ_c (strain induced yielding) with ϕ at two different temperatures is shown in Figure 7 (f). It is observed that γ_c is decreasing with increasing ϕ . For example, the value of γ_c for 0–10% is one order of magnitude larger than that of $\phi = 20\%$. This behaviour can be explained based on the jammed domains of particles as seen in the rheomicroscopy (Figure 5, SmA phase). With increasing ϕ , the number density and size of these jammed domains increase. As they are fragile, under low strain amplitude, they can break and flow; as a result, the critical strain amplitude (γ_c) is expected to decrease [32].

It is known that the colloidal suspension can undergo glass transition with increasing particle concentration and can crystallise under favourable



Figure 8. (Colour online) Angular frequency dependence of storage (G') and loss (G'') moduli at different volume fractions at constant strain amplitude 0.03% at $T = 25^{\circ}$ C. Continuous lines are the best fit to Equation (1). Vertical arrows indicate the minimum in G''.

condition [32,33]. To see this aspect, we studied the frequency dependence of the storage (G') and loss (G'') moduli. Figure 8 shows the angular frequency dependence of G' and G'' at different volume fractions.

The SmA phase (without colloids) shows typical frequency dependence, i.e. both moduli are increasing with frequency. noted It is that for $\phi = 1\%, 5\%$ and $10\%, G''(\omega)$ exhibits a shallow minimum. This is due to the increase in $G''(\omega)$ at both lower and at higher frequencies. The low frequency rise is due to a very slow structural rearrangement of the particles suggesting that there exists a relaxation process. The high-frequency rise signifies the

domination of viscous relaxation of fluid phase [34,35]. This is also a typical characteristic of many colloidal soft glasses. Hence, we analyse the results based on the soft glassy rheology model. In this model, the real part of the storage modulus is given by Refs [36,37]:

$$G'(\omega) = G_0 + \beta \omega^{\alpha} \tag{1}$$

where G_0 is the plateau modulus that arises due to the static defects in SmA and the frequency-dependent term results from the bulk response of the materials. β arises from the disorientated (misaligned) parts of the sample [26,36,38]. The fit parameters are plotted with volume



Figure 9. (Colour online) (a) Variation of α and the ratio G''/G' as a function of volume fraction of particles ϕ . (b) Variation of G_0 and β with ϕ . The red line is the best fit result $G_0 \sim \phi^{1.8 \pm 0.2}$.

fraction in Figure 9. It is observed that α decreases with increasing volume fraction and tends to zero ($\alpha \rightarrow 0$) up to $\phi = 10\%$ (Figure 9(a)). Similar behaviour was also observed in many colloidal soft glass materials [26,33]. The variation of the ratio G''/G' with ϕ is also shown in Figure 9(a). We observe that $G''/G' \sim \alpha$ as expected in the soft glassy rheology model [26,37,39]. Figure 9(b) shows the variation of G_0 and β with volume fraction. The increase of β with ϕ indicates that the contribution from the disoriented parts of the sample increases due to the inclusion of the particles. The increase of G_0 indicates that the smectic line defects dominate this soft glassy rheology. Theoretically in analogy with rubber elasticity, the elastic response of static defect networks should vary as $G_0 \sim \rho^2$, where ρ is the density of the particles [26,36]. In our experiment, we find that $G_0 \sim \phi^{1.8 \pm 0.2}$ (Figure 9 (b)). Similar exponent was also reported in case of aerosildispersed SmA LCs (8CB) [26]. At higher concentrations $(\phi = 20\%)$, both α and the ratio G''/G' tend to increase with ϕ . We conjecture that at this high-volume fraction of particles under oscillation, some short-range order among the particles is developed. However, further study is required.

4. Conclusion

In conclusion, we have studied the rheological properties of nematic and SmA LC colloids prepared by dispersing spherical microparticles with homeotropic boundary condition in 8CB LCs. The microparticles create chains mediated by the induced defects with quadrupolar director configuration and form networks with increasing volume fraction. We observed a shear-thickening behaviour in the low shear rate range. The rheomicroscopy studies reveal the shear rate-dependent distinct dynamics of the networks. The dynamic response of the SmA colloids shows a soft glassy rheology with increasing volume fractions up to a certain range. Finally, this study shows that the rheological properties of nematic and SmA colloids are significantly contributed by the particle-induced defects and their self-assembly.

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Disclosure statement

No potential conflict of interest was reported by the authors.

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