

Rheology and structure of worm-like micelles

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Abstract

Although worm-like micelles have been studied for over 20 years, the diversity of macroscopic behavior and the potential analogy to polyelectrolytes has driven continued study. During the last year, development and application of more realistic scattering models has yielded a deeper understanding of micelle structure. Comprehensive studies on systematic systems are being reported that combine structural and macroscopic data. These studies provide the fundamental understanding necessary to quantify the coupling between micelle structure and rheology. Finally, with increased understanding of these systems, there is a growth in the number of novel applications of worm-like micelles. © 2001 Elsevier Science Ltd. All rights reserved.

Keywords: Micelles; Worm-like; Rod-like; Thread-like; Rheology; Scattering

1. Introduction

Micelles tend toward elongated structures when the packing parameter, $p = v/Al$, of the surfactant increases towards $p = 1/2$, where v is the volume of the hydrophobic part of the surfactant, A is the surface area occupied by the surfactant head group and l is the extended length of the hydrophobic portion [1]. Micelles are considered rod-like if the length of the micelle is short compared to its persistence length (the length over which it is rigid), and worm- or thread-like when the overall length, or contour length, is much greater than its persistence length. An analogy is drawn between worm-like micellar systems and polyelectrolytes, although micelles have the additional ability to break and reform, gaining the moniker ‘equilibrium polymers’ [2]. The dynamics of these systems are of great interest, because

subtle changes in the surfactant, counterion and added electrolyte alter the dimensions, flexibility and interactions of the micelles, leading to marked effects on the macroscopic rheological behavior.

These systems already find uses as viscosity enhancers, but as our understanding increases, new applications form. Recently, worm-like micelles have been considered as a novel sieving matrix for the separation of DNA fragments by capillary electrophoresis [3], templates for asymmetric and aligned nanostructures [4–6] and as drag-reduction additives in district heating systems [7]. In all of these applications, knowledge of the structure and dynamics of the worm-like micellar systems is vital for optimization of the process.

Whether for the sake of fundamental understanding, as model polyelectrolytes, or to develop new applications, a detailed understanding of the relationship between composition, micellar structure and macroscopic rheology is vital. For this, tools are required to characterize micelle morphology and dynamics, and models are needed to relate these to macroscopic behavior. Advances in each of these have been made in the last few years.

Abbreviations: CTAB, cetyltrimethylammonium bromide; CTAT, cetyltrimethylammonium tosylate; KBr, potassium bromide; NaSal, sodium salicylate.

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2. Structure

The structural length scales of worm-like micelles, radius, persistence length and contour length, are often disparate. Radii are typically a few nm, persistence lengths are 10s of nm and contour lengths range from 100s of nm to μm and larger. This disparity allows modeling of scattering to be broken down into different regimes and attacked independently. This fact, along with the observation that surfactants scatter much more strongly than polymers in both neutron- and light-scattering, has given rise to the suggestion that these are model systems to explain and study polyelectrolyte behavior [2,8].

Early scattering models treated neutral chains with contour lengths much greater than the persistence length. Recent advances allow non-homogeneous and non-axisymmetric radial cross-sections to be extracted [9•], the excluded volume of the chains to be taken into account [10•], electrostatics to be incorporated into the persistence length via existing theories for polyelectrolyte [8,10•,11] and intermicellar interactions to be included for rod-like micelles [12]. This year has seen some of the first full fits of scattering data from charged systems of worm-like micelles to extract flexibility as a function of concentration and ionic strength [13•]. A recent study extended structural characterization to systems more than 65-fold greater than the overlap concentration, with added screening electrolyte [9•,13•].

Uncharged systems still exhibit pertinent areas of research, as recent results on the change in micellar dynamics at high pressures demonstrate [14]. Given that structural analysis of weakly charged systems can now be performed with confidence, whole new systems and behavior can be tackled. Comparison to experimental data provides realistic measures of persistence length and its dependence on concentration and ionic strength. For a wide variety of systems, persistence lengths range between 20 and 40 nm [9•], although recent measurements of a system of sugar surfactant, hexanol and water is much more flexible, with persistence length closer to 10 nm [11]. Re-evaluation of previous light-scattering studies showed that earlier reports of much larger persistence lengths are an artifact of neglecting intermicellar interactions in the analysis [9•].

The packing parameter is a strong function of counterion and electrolyte, and several reports demonstrate these effects. Relatively small changes in the structure of penetrating anionic counterions to trimethylammonium-based surfactants show the variation in structural transitions of these systems [15–18•]. Systematic studies to investigate counterion and, more important, electrostatic effects have been initiated using mixtures of cationic/anionic sur-

factants [19••], variation of counterion inventory through electrolyte composition [20,21], ‘doping’ of non-ionic micelles with ionic co-surfactant [8,10•] and the use of ionizable surfactants based on tetradecyldimethylamine [9•]

Improvements in the resolution of cryo-transmission electron microscopy (TEM) have brought about an increased number of direct structural probes of worm-like micellar systems. Studies have demonstrated that ‘endcaps’ have a larger diameter than the cylindrical body in Gemini surfactants [22•] and characterized the transitions from worm-like micelles to branched systems [23]. While cryo-TEM is a powerful tool in the characterization of structure, the danger of shear during sample preparation has been demonstrated and is a concern when working with shear-sensitive structures [24•,25].

Attempts to capture the structure of worm-like micelles in the bulk have led to the formation of novel aggregates [4,5]. By polymerizing the penetrating counterion in worm-like micelles based on the cationic surfactant, polyelectrolyte–surfactant aggregates are generated. The structural changes during polymerization are sufficiently complex that the length of the micelles is not maintained during polymerization, but the cross-section is captured and the potential for generating novel particles exists.

3. Linear rheology

As the contour length increases, worm-like micelles become entangled. A competition between reptation and the breaking/reforming time of the micelles gives rise to simple linear rheological behavior, often referred to as ‘Maxwell-like’ [26]. The linear behavior is described by two parameters — a plateau modulus (G_0) related to the entanglement density of the mesh and a relaxation time (τ_R). This dominant relaxation time is simply the geometric mean of the reptation and breaking/reforming times of the micelles. With this concise description of viscoelasticity, a goal for this field is to predict τ_R and G_0 from knowledge of the local micellar structure.

Many of the structural studies mentioned above have also included characterization of the linear rheology [18•,19••,27–29]. Since the relationship between persistence length and rheology is not known, we cannot predict rheology a priori. However, there has been success in scaling the linear rheological properties with concentration [19••,27,28,30]. A transformation from linear micelles to a branched system has been quantified in several systems by observing increased fluidity [18•,19••,31,32]. Cryo-TEM has also shown signs of branched systems, in agreement with rheological measurements [22•,23]. The relationship

between micelle stiffness and the onset of branching is of considerable importance and a salient avenue of study [18•].

Mechanical rheometry possesses problems of instrument inertia and environment control, but linear rheology can also be probed with microrheometry. The Brownian motion of a colloidal polystyrene sphere in worm-like CTAB/KBr solutions has been probed using diffusing wave spectroscopy and the Maxwell relaxation parameters extracted [33].

Application of the single relaxation-time Maxwell model to more complex or incomplete frequency data is dangerous. The loss modulus deviates from simple Maxwell behavior at higher frequencies as the ‘breathing modes’ or breaking and reforming time scales influence the relaxation spectrum. As these modes move closer to the dominant Maxwell relaxation time, the extraction of a single value of τ_R is problematic. The inversion of the frequency data to extract relaxation times is inherently ill-posed; this problem has long been considered in extracting the relaxation spectrum for polymer solutions and melts from dynamic data. As more detailed analysis of the dynamic data to extract multiple and coupled relaxation times continues (see for example [30]), these analytical techniques will need to be considered. It is important to note that the high-frequency upturn in the loss modulus must be characterized. While there is potential to use this break point to characterize the persistence length of the micellar mesh [27], if the frequency at which this is observed is close to the dominant relaxation time, then the assumptions of a ‘fast-breaking’ micellar mesh is questionable — fitting a model solely to the low frequency and cross-over region will inadvertently omit pertinent dynamics.

4. Non-linear rheology

At shear rates high enough to disturb the equilibrium structure, non-linear rheological responses are observed. Worm-like micellar systems exhibit a rich rheology, with many distinct phenomena not observed in other macromolecular species. Dilute solutions of cationic micelles exhibit pronounced shear-thickening under simple shear [34,35]. Semi-dilute micellar solutions that exhibit model viscoelastic behavior in the linear regime often exhibit stress plateaus and shear banding at higher shear rates [36]. Neither phenomenon can be predicted a priori, and both (and the possible link between the two) are active areas of research.

The increase in steady-state viscosity with applied rate, or shear thickening, is primarily observed in dilute cationic surfactants with penetrating counterions at low levels of added electrolyte. Studies on

shear thickening of the cetyltrimethylammonium tosylate system [37] have continued by probing transient rheology and small-angle neutron scattering [38,39]. The mechanism proposed for shear thickening is that this system undergoes shear-induced micellar growth and a transition from rod-like micelles to aligned worm-like micelles. By interpreting the peak in the scattered intensity as a correlation distance, the authors show that shear moves the position of the scattering peak to lower values of the scattering vector, q , and towards the $q \sim \phi^{1/2}$ relation expected for entangled worm-like objects [39]. An independent in situ measure of length would be necessary to verify this conclusion. Similar rheological and scattering results have been observed recently in Gemini surfactants [40•]. Both of these groups have demonstrated the sensitivity to temperature and sample history [38,40•]. Dilute solutions of mixtures of cationic and anionic surfactants show shear thickening at distinct ratios of the oppositely charged surfactants, providing an alternate mechanism to characterize the influence of micellar-surface charge density [19••]. Uncharged micelles of the phospholipid lecithin suspended in *n*-decane and swollen with water show shear thickening, but the transient response is significantly different, so it is unclear if this is a shear-induced transition or a restructuring of the gel [41].

The addition of non-ionic polymer allows for control of the critical shear rates and the viscosity [42]. Subtle changes in the structure of the polymer allow the shear thickening to be enhanced or eradicated completely. Characterization of the specific polymer–surfactant interactions will allow understanding of the transition.

Two recent papers have suggested counterion-mediated attraction as the cause of the shear-induced structures [40•,43••]. A model has been developed that predicts the critical shear rates and temperature dependence observed experimentally [43••]. While this approach arises from existing work in like-charged rod-like colloids and polyelectrolytes, testing theories with worm- and rod-like micelles is complicated by the dependence of length and persistence length on temperature, salt and concentration. However, a carefully constructed study could provide structural and rheological data necessary to test the prediction of shear-induced bundling.

The shear-induced alignment of CTAB/NaSal solutions is used to template the gelation of silica to create highly aligned nanostructures [6]. The resulting structures formed are clearly more aligned in systems that exhibit shear thickening. Some cationic surfactants are shown to be good drag reduction agents, but the mechanism is not understood. A recent series of papers attempts to correlate solution viscoelasticity, elongational flow behavior and structure with drag

reduction. The influence of counterion in cationic systems [7,44,45], ratio of surfactant in mixtures of cationic amphiphiles of varying alkyl chain length [46], added salt in cationic systems [47] and ratio of cationic to zwitterionic surfactants [48] are probed. The prerequisite for drag reduction appears to be the presence of thread- or worm-like micelles. Cryo-TEM, the primary microstructural probe in these studies, might indicate the requirement of a shear-sensitive structure, but the range of pertinent deformation rates is still in question.

Shear banding is observed in many worm-like micellar solutions in the semi-dilute region. Several mechanisms are proposed for shear banding, and tools are still being developed to properly characterize structures that develop in a non-homogeneous flow field. Again, the behavior is dependent on the equilibrium structure of the micelles. For CTAB in D₂O at concentrations near an isotropic-to-nematic transition concentration, a shear-induced transition to a viscous nematic band and lower-viscosity isotropic band is verified using NMR [49•]. Results agree with neutron scattering measurements of total alignment and a simple model, but the existence of a highly viscous nematic phase still needs to be explained. For systems at concentrations far from phase boundaries, the phenomenon is being investigated as a constitutive anomaly — the existence of multivalued points in the stress–strain relationship. A model based on a co-deformational Maxwell model (similar to a transient network model) quantitatively captures the steady state and transient rheology observed in concentrated CTAT solutions [50]. In another system, optical and mechanical rheological studies on isotropic worm-like micelle solutions quantify the kinetics of this transformation [51•]. A system of CTAB with the inorganic salt sodium nitrate shows a stress plateau at higher shear rates, no hysteresis in the nonlinear rheology and adherence to the stress–optic relation up to the point of shear banding. Transient rheology shows a fast response from the viscoelastic solution and a slow response thought to be associated with the formation and growth of the shear bands. However, optical measurement of the birefringence across the gap shows that the shear bands are still stabilizing long after the rheological signals stabilize [51•].

Use of the seemingly model linear viscoelastic behavior in studies of complex flows have been hindered by the aberrant non-linear behavior. However, a recent report of the wake dynamics of a gas bubble rising through a 10.7 mM equimolar CTAB/KBr solution shows instabilities [52]. The sensitivity of this phenomenon to viscoelasticity might be used to understand the non-linear and elongational properties of worm-like micelles, a topic only briefly attacked in the literature [53,54].

5. Conclusions

As systems become better characterized, both the structural and macroscopic behavior can be quantified. This has already begun with the reverse lecithin micelles (organogels) that are being probed rheologically, even with an applied electric field [30,41,55]. These systems are neutral, so we do not expect the same breadth of behavior observed in the low-salt cationic or other charged systems, particularly the formation of shear-induced structures. Candidates for modeling polyelectrolyte behavior are the mixed cationic/anionic systems [19••], the non-ionics ‘doped’ with ionic co-surfactants [8,10•] and systems in which the composition of the surface counterions is characterized [20,21].

Challenges for the future include characterization of the overall length of worm-like micelles in charged systems and systems in which the pertinent length scales overlap. As physical modeling of the rheological phenomenon becomes tractable, development of techniques for in situ characterization of aligned structures under flow will become essential.

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