

Wormlike emulsion droplets

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Forming an interface between immiscible fluids incurs a free-energy cost that usually favors minimizing the interfacial area. An emulsion droplet of fixed volume therefore tends to form a sphere, and pairs of droplets tend to coalesce. Surfactant molecules adsorbed to the droplets' surfaces stabilize emulsions by providing a kinetic barrier to coalescence. Here, we show that the pressure exerted by bound surfactant molecules also competes with the droplet's intrinsic surface tension and can reverse the sign of the overall surface free energy. The onset of negative surface tension favors maximizing surface area and therefore favors elongation into a wormlike morphology. Analyzing this system in the Gibbs grand canonical ensemble reveals a phase transition between spherical and wormlike emulsions that is governed by the chemical potential of surfactant molecules in solution. Predictions based on this model agree with the observed behavior of an experimental model system composed of lipid-stabilized silicone oil droplets in an aqueous surfactant solution.

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I. INTRODUCTION: WORMLIKE EMULSIONS

The droplets in oil-in-water emulsions generally minimize their interfacial area to lower their free energy. This mechanism favors the formation of spherical droplets and promotes droplet coalescence. Droplets can be kinetically stabilized against coalescing by binding surfactant molecules to the oil-water interface. Here, we describe an additional dramatic consequence of surfactant binding, illustrated in Fig. 1, in which the pressure exerted by bound molecules drives a spherical emulsion droplet through a phase transition to a wormlike morphology. Wormlike emulsions are distinct from wormlike micelles [1,2] because they are stabilized by surfactant concentrations far below the critical micelle concentration (CMC). They also differ from the varied morphologies of liposomes and vesicles, which are dictated by the bending and curvature moduli of surfactant bilayers [3] and bicontinuous emulsions that are stabilized by colloidal particles trapped at the oil-water interface [4,5]. The sphere-to-worm transition in suitable emulsions constitutes an alternative pathway for morphology control in soft matter.

Our study of wormlike emulsions is inspired by the observed behavior of micrometer-scale droplets of a ternary oil mixture stabilized by lipid surfactants and dispersed in water [6]. This model system has been studied extensively as a building block of emulsion-droplet chains known as colloidomers [7]. Individual droplets ordinarily form spheres, as shown in Fig. 1(a). We find that adding a cosurfactant, such as sodium dodecyl sulfate (SDS), can cause these droplets to elongate into flexible wormlike structures [8] such as the example in Fig. 1(b). Spherical droplets are observed to deform continuously into worms when their dispersion is cooled

a few degrees, and reform into spheres upon heating. The transformation is reversible through repeated cycles. Simple linear worms result from quasistationary cooling and more complex morphologies emerge at higher cooling rates.

II. MINIMAL MODEL FOR THE SPHERE-TO-WORM TRANSITION

The cosurfactant appears to play an essential role in the sphere-to-worm transition because our system undergoes no transformations in the absence of added SDS. We therefore propose a minimal model, depicted schematically in Fig. 2, that explains how surfactants can mediate morphological transformations in emulsion droplets. This model treats an oil droplet as the substrate for a two-dimensional classical gas of surfactant molecules. Each surfactant molecule has mass m and occupies an area Ω on the droplet's surface. Surfactant molecules bind to the surface with a binding energy ϵ that we assume to be independent of coverage. A system of N bound molecules therefore has the Hamiltonian

$$H_N = \sum_{j=1}^N \frac{p_j^2}{2m} - N\epsilon, \quad (1)$$

where $\mathbf{p}_j$ is the two-dimensional momentum of the j th molecule at location $\mathbf{r}_j$ on the droplet's surface. The associated canonical partition function is [9]

$$Z(A, T, N) = \frac{1}{N!} \frac{1}{h^{2N}} \int e^{-\beta H_N} \prod_{j=1}^N d^2 r_j d^2 p_j \quad (2a)$$

$$= \frac{1}{N!} \frac{1}{\lambda^{2N}} \left(A - \frac{1}{2} N \Omega \right)^N e^{N\beta\epsilon}, \quad (2b)$$

where h is Planck's constant, $\beta^{-1} = k_B T$ is the thermal energy scale at absolute temperature T , A is the droplet's surface area, and

$$\lambda(T) = \frac{h}{\sqrt{2\pi m k_B T}} \quad (3a)$$

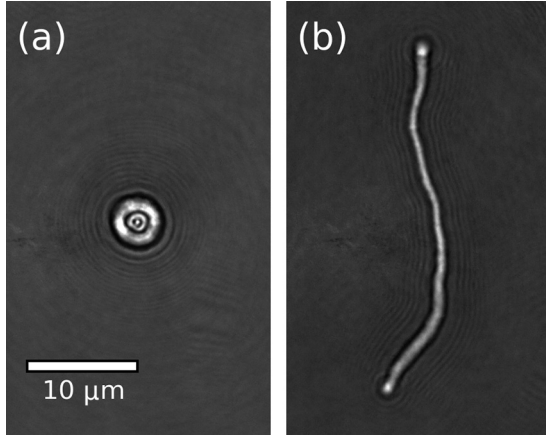


FIG. 1. Holographic images of a 3.2- μm -diameter lipid-stabilized emulsion droplet composed of a ternary oil mixture, dispersed in an aqueous SDS solution at 1 mM: (a) in the spherical morphology at $(23 \pm 1)^\circ\text{C}$; and (b) in the wormlike morphology [8] at a slightly lower temperature, $(21 \pm 1)^\circ\text{C}$.

is the bound molecules' thermal de Broglie wavelength [10]. The term in parentheses on the right-hand side of Eq. (2b) accounts for the area on the droplet's surface that is occupied by surfactant molecules [9].

Bound surfactant molecules are in thermodynamic equilibrium with the bulk solution of free surfactant molecules at chemical potential μ . In establishing this equilibrium, the two-dimensional pressure of the molecules bound to the surface favors increasing the droplet's surface area by deforming the droplet into a noncompact shape such as a worm. Increasing the surface area, however, incurs a cost in surface energy through the fluid's intrinsic surface tension, σ . We account for changes in the droplet's surface area and surfactant coverage with the Gibbs grand canonical partition function,

$$\mathcal{Q}(\sigma, T, \mu) = \int_{A_0}^{A_1} \frac{dA}{\lambda^2} \sum_{N=0}^{N_{\max}} \mathcal{Z}(A, T, N) e^{\beta N \mu} e^{-\beta \sigma A}, \quad (4)$$

where A_0 is the minimal surface area of the bare spherical droplet, and A_1 is the maximum area of the deformed wormlike droplet. The minimum area is set by the droplet's volume. The maximum area is limited by such factors as the disjoining pressure [11,12] and short-ranged interactions among the lipid surfactants [1] that set a lower limit on the droplet's minimal dimension. Assuming that surfactant molecules do not overlap, close packing constrains the upper limit of the sum in Eq. (4) to $N_{\max} = A/\Omega$.

Substituting Eq. (2b) into Eq. (4) and rearranging terms allows us to recast the Gibbs grand partition function,

$$\mathcal{Q}(\sigma, T, \mu) = \int_{A_0}^{A_1} \frac{dA}{\lambda^2} \mathcal{Z}(A, T, \mu) e^{-\beta \sigma A}, \quad (5)$$

in terms of the grand canonical partition function,

$$\mathcal{Z}(A, T, \mu) = \sum_{N=0}^{N_{\max}} \frac{1}{N!} \left(\frac{A - \frac{1}{2} N \Omega}{\lambda^2} \right)^N e^{\beta N(\epsilon + \mu)}. \quad (6)$$

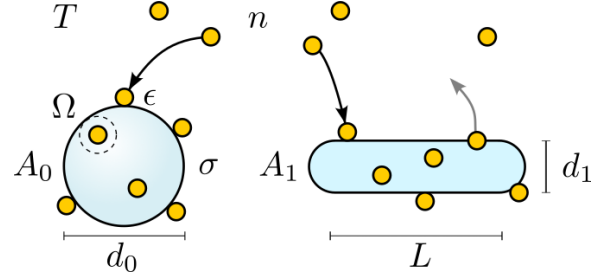


FIG. 2. Schematic representation of surfactant molecules (yellow) binding to a spherical emulsion droplet, thereby inducing it to adopt a wormlike morphology.

For simplicity, we assume that the coverage is small enough that we may neglect the excluded area. In this approximation, the grand canonical partition function reduces to

$$\mathcal{Z}(A, T, \mu) \approx \sum_{N=0}^{\infty} \frac{1}{N!} \left[\frac{A}{\lambda^2} e^{\beta(\epsilon + \mu)} \right]^N \quad (7a)$$

$$= \exp \left(\frac{A}{\lambda^2} e^{\beta(\epsilon + \mu)} \right). \quad (7b)$$

The Gibbs grand canonical partition function then follows as

$$\mathcal{Q}(\sigma, T, \mu) \approx \int_{x_0}^{x_1} \exp(x[e^{\beta(\epsilon + \mu)} - \beta \sigma \lambda^2]) dx \quad (8a)$$

$$= \frac{e^{-\kappa x_0} - e^{-\kappa x_1}}{\kappa}, \quad (8b)$$

where $x = A/\lambda^2$ is the dimensionless surface area and

$$\kappa(T, \mu) = \beta \sigma \lambda^2(T) - e^{\beta(\epsilon + \mu)} \quad (9)$$

is a dimensionless parameter that describes competition between the intrinsic interfacial tension and the two-dimensional pressure of the bound surfactants. The sign of $\kappa(T, \mu)$ selects the droplet morphology and is determined by the temperature of the system and the chemical potential of the surfactant molecules.

Treating unbound surfactant molecules as an ideal gas with a bulk number density n , the chemical potential is given by the Sackur-Tetrode formula [14,15],

$$\mu = k_B T \ln(n \lambda^3). \quad (10)$$

Applying this to Eq. (9) suggests that $\kappa(T, \mu)$ vanishes for values of the surfactant concentration that fall along a curve, $n = n_c(T)$, where

$$\frac{n_c(T)}{n_0} = \left(\frac{T_0}{T} \right)^{\frac{1}{2}} \exp \left(-\frac{T_0}{T} \right). \quad (11)$$

This curve demarks the phase boundary between the spherical and wormlike morphologies and is plotted in Fig. 3(a). The temperature scale in Eq. (11) is set by the surfactant's binding energy through

$$T_0 = \frac{2\epsilon}{k_B}, \quad (12)$$

while the characteristic concentration scale,

$$n_0 = \sqrt{\frac{2\pi m \sigma^2}{h^2}} \frac{1}{\epsilon}, \quad (13)$$

balances binding energy against surface tension.

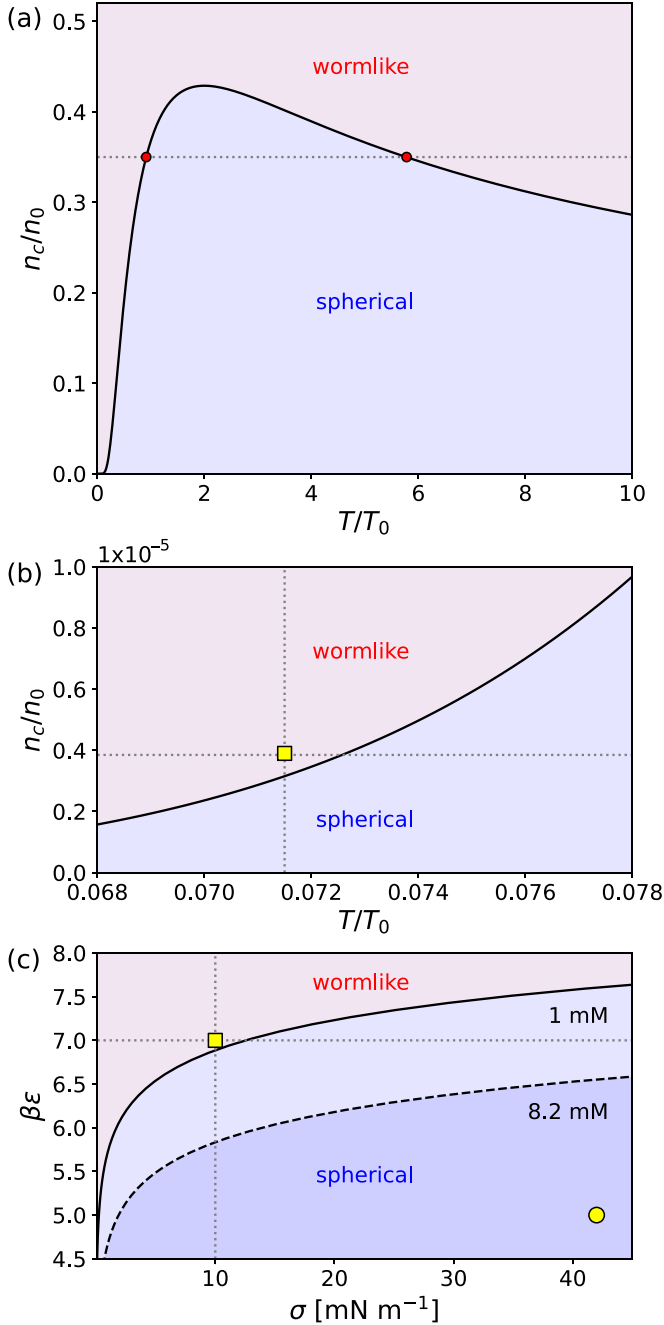


FIG. 3. (a) Phase boundary between spherical and wormlike morphologies predicted by Eq. (11) as a function of temperature T and surfactant concentration n . (b) Region of the phase diagram around the experimental conditions in Fig. 1, which are plotted as a discrete (square) point. Proximity to the predicted phase boundary is consistent with the system's sensitivity to temperature changes. (c) Phase boundary plotted in terms of the intrinsic interfacial energy σ of the droplets and the binding energy for SDS, ϵ , in an SDS solution at 1 mM (solid curve) and at the CMC for SDS (dashed curve). Properties for the ternary oil droplets used for this study fall near the phase boundary at 1 mM (square). Properties for standard silicone oil in SDS solution [13] (circle) lie far from the phase boundary, consistent with observation.

The effective surface tension $\kappa(T, \mu)$ is positive in the absence of dissolved surfactant molecules, $n = 0$, which is

consistent with the expectation that emulsion droplets ordinarily form spheres. Conversely, $\kappa(T, \mu)$ is strictly negative when the concentration of dissolved surfactant molecules exceeds $n^* = n_0/\sqrt{2e} = 0.429 n_0$. Between these two limiting cases, the sign of the effective surface tension depends on the temperature, with spherical droplets being stable in a range, $T_- < T < T_+$, that is set by the bulk surfactant concentration.

The average surface area adopted by a droplet can be obtained in the Gibbs grand canonical ensemble,

$$\langle A \rangle = -\frac{1}{\beta} \frac{\partial}{\partial \sigma} \ln \mathcal{Q} \quad (14a)$$

$$= \frac{\lambda^2}{\kappa} \frac{(\kappa x_0 + 1)e^{-\kappa x_0} - (\kappa x_1 + 1)e^{-\kappa x_1}}{e^{-\kappa x_0} - e^{-\kappa x_1}}. \quad (14b)$$

Assuming that $|\kappa|x_1 \gg |\kappa|x_0 \gg 1$, the sign of κ selects either spheres or worms:

$$\langle A \rangle \approx \begin{cases} A_0, & T_- < T < T_+ \Rightarrow \text{spherical}, \\ A_1, & \text{otherwise} \Rightarrow \text{wormlike}. \end{cases} \quad (15)$$

The nature of the surfactant-mediated phase transition is clarified by considering the mean number of surfactant molecules that are bound to the droplet:

$$\langle N \rangle = \frac{1}{\beta} \frac{\partial \ln \mathcal{Q}}{\partial \mu} = \frac{\langle A \rangle}{\lambda^2} e^{\beta(\epsilon + \mu)}. \quad (16)$$

This corresponds to a fractional surface coverage,

$$\phi(n, T) \equiv \Omega \frac{\langle N \rangle}{\langle A \rangle} = n \Omega \lambda e^{\beta\epsilon}, \quad (17)$$

that depends strongly on temperature. High surfactant coverage in the low-temperature phase provides the pressure needed to overcome the droplet's intrinsic surface tension and destabilize the spherical morphology. Equation (17) also can be used to assess whether or not the surfactant's excluded area can be neglected in deriving Eq. (7). Taking the excluded area into account tends to reduce the number of bound surfactant molecules while also tending to increase their surface pressure. These competing effects influence the phase boundary $n_c(T)$ quantitatively but not qualitatively.

III. EXPERIMENTAL OBSERVATIONS

The predicted sphere-to-worm transition can be compared directly to the observed behavior of emulsion droplets, such as the example in Fig. 1. The droplets used in this study are synthesized from a 1:1:1 mixture by volume of three silane monomers: (a) dimethyldiethoxysilane (Sigma-Aldrich), (b) (3,3,3-trifluoropropyl) methyldimethoxysilane (Gelest), and (c) 3-glycidoxypopylmethyldiethoxysilane (Gelest). This oil is formed into micrometer-scale droplets by ammonia-catalyzed hydrolysis followed by condensation, as described in Refs. [16,17]. Variations on this protocol reproducibly yield droplets with diameters ranging from 1 to 10 μm [16]. The droplets are cleaned and dispersed in 1 mM SDS solution to an overall concentration of 1×10^7 droplets/mL before being stabilized by adding DSPE-PEG-SH lipid (Avanti Polar Lipids, MW 2000) at a concentration of 5 $\mu\text{g mL}^{-1}$. This procedure yields droplets with an estimated surface

coverage of lipids of roughly 50% [17]. Lipids bind irreversibly to the oil-water interface [18] and are not observed to form micelles [19,20]. Because the number of bound lipid molecules does not change once the droplets are formed, lipids reduce the droplets' intrinsic interfacial tension σ but do not otherwise contribute to the morphological transition.

The 1 mM concentration of SDS in our model system phenomenologically is close to the point at which spherical droplets deform into worms. The transformation is not observed in dispersions that lack SDS. Each SDS molecule has a mass of $m = 4.8 \times 10^{-25}$ kg and occupies an area $\Omega \lesssim 0.5$ nm² on the oil-water interface [21]. The interfacial tension of the oil-lipid-water system is measured with a pendant drop tensiometer (Attension, Theta Lite) to be $\sigma = 10$ mN m⁻¹ in the absence of SDS [6], which is consistent with values obtained for similar systems in previous studies [22–24]. The dependence of the surface energy on SDS concentration yields $\epsilon = 7k_B T$ for the surfactant binding energy [6,23]. Equation (17) then yields $\phi \lesssim 2 \times 10^{-3}$ for the fractional surface coverage of SDS at room temperature, which is small enough to justify neglecting Ω in the derivation of $\mathcal{Z}(A, T, \mu)$, but still large enough to drive the transition.

Substituting the same material parameters into Eq. (13) yields $n_0 = 256$ M for the characteristic concentration scale of SDS. This value is used in Fig. 3(b) to replot the phase diagram in the vicinity of the experimental conditions at $n = 1$ mM and $\beta T_0 = 14$. This comparison confirms that the model system's properties lie close to the predicted phase boundary.

Figure 3(c) recasts these results in terms of the values of the surface tension and binding energy that lie along the phase boundary for a specified surfactant concentration. The solid curve presents the values of ϵ and σ that would satisfy $n = n_c(T)$ at the experimental SDS concentration, $n = 1$ mM. This again confirms that the material properties of the model system are close to the predicted transition point in a 1-mM SDS solution at room temperature. Conventional silicone oils, by contrast, have substantially higher surface tension in contact with water and lower affinity for SDS [13], and therefore lie well within the predicted domain of stability for spherical droplets. Such systems cannot be destabilized into wormlike droplets by increasing the surfactant concentration because the CMC for SDS at 8.2 mM establishes an upper bound on the chemical potential of bound molecules at the interface. The phase boundary at that concentration is plotted as a dashed curve in Fig. 3(c). The ubiquity of such limits may explain why wormlike emulsions have not been reported previously.

We analyze emulsion droplets' behavior using a custom-built instrument that combines conventional bright-field microscopy, in-line holographic microscopy, and holographic optical trapping [25]. The microscope uses an oil-immersion objective lens (Nikon S Plan Apo, 100 $\times$ numerical aperture 1.4) to achieve a system magnification of 48 nm/pixel on the sensor of a CMOS video camera (Flir Flea3 USB3.0). Holographic imaging is performed at a vacuum wavelength of 447 nm (Coherent Cube) and an intensity of 1 mW mm⁻². Holographic trapping is performed at 1070 nm (IPG Photonics YLR-10-LP) using a phase-only spatial light modulator

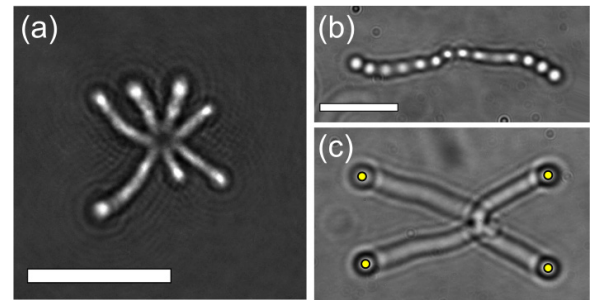


FIG. 4. (a) Holographic image of a multibranched wormlike emulsion droplet formed by rapid cooling [8]. (b) Bright-field image of a wormlike emulsion droplet undergoing a pearling instability. (c) Bright-field image of two wormlike emulsion droplets crossed under tension by optical tweezers (yellow circles) projected onto their ends. Scale bars: 10 μ m.

(Holoeye Pluto). The images in Fig. 1 are holograms of the emulsion droplet that can be used to numerically reconstruct the deformed droplet's three-dimensional structure [26] and to track its motion [27].

The ramified structure in Fig. 4(a) is a wormlike emulsion droplet formed from a spherical droplet by cooling the microscope stage 3 $^{\circ}$ C in 1 min. Rather than continuously elongating into a uniaxial worm, this rapidly cooled droplet nucleated seven wormlike extensions. Even though the individual arms are flexible [8], they do not relax into a uniaxial worm resembling Fig. 1(b) presumably because pressure exerted by the two-dimensional gas of bound surfactant molecules tends to keep them all extended. The formation and stability of such starlike worms therefore provides additional support for the proposed morphology selection mechanism. Multiarm droplets contract into spheres upon heating and can be thermally cycled into either uniaxial or ramified worms through the choice of cooling rate.

Rapidly heating a linear worm can induce a pearling instability of the kind depicted in the bright-field image in Fig. 4(b). In this case, the sample's temperature was raised by 3 $^{\circ}$ C over 1 min, which places it roughly on the predicted phase boundary. The pearling instability does not cause the droplet to break up but does appear to hinder its return to sphericity; such droplets require an hour or more to relax into spheres. The delay may be caused by stress-induced redistribution of lipid surfactants on the droplet's surface.

The image in Fig. 4(c) highlights wormlike droplets' stability against coalescence. Two droplets are trapped by holographic optical tweezers that are projected onto their ends. The droplets' axes are made to intersect by moving the traps interactively in three dimensions. The droplets then are placed under an estimated 100 fN of tension by moving the tweezers apart. Each micrometer-diameter trap is powered by 10 mW, and the intense illumination raises the local temperature by roughly 1 $^{\circ}$ C. This localized heating causes the ends of the worms to form spherical bulges that are visible in Fig. 4(c). Despite 2 min of heating and the imposed tension, the two worms do not merge. Extinguishing

the optical tweezers allows them to separate and to diffuse independently.

IV. DISCUSSION

The observations reported in Fig. 4 lend credence to the proposal that the sphere-to-worm transformation is a phase transition mediated by binding of SDS cosurfactant at constant chemical potential, and that the extended droplets are protected from either breaking up or coalescing by the lipid surfactant. These observations further demonstrate that the nature of the morphological transformation can be controlled kinetically, with different morphologies appearing at different cooling and heating rates. The discovery of wormlike emulsions in our model system raises the possibility that similar shape-shifting transformations will be found in other emulsions. The ease with which the sphere-to-worm transformation can be controlled both macroscopically and microscopically suggests that this class of emulsions

will be useful for developing active and responsive soft materials [28–31].

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DATA AVAILABILITY

The data that support the findings of this article are openly available [32].

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