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Cryo-Electron Microscopy: Attempts to Watch the Formation of Dilute Emulsion via Microemulsion

Hanseung Lee[1], Eric D. Morrison[2], and Alon V. McCormick[1]

[1.] Chemical Engineering and Materials Science, University of Minnesota, Twin Cities, Minneapolis, MN 55455
[2.] Segetis, Golden Valley, MN 55427

The dilute emulsions are ubiquitous in our environments. These colloidal systems have found widespread use in our lives such as foods, consumer products, and industrial applications for detergency. [1] Thus, it is important that we understand how to formulate the colloidal particles and precisely control their structures. In addition, the emerging nanometer-scale emulsion system will eventually offer more opportunity for conventional emulsion industries.

However, monitoring nanostructures in these complex fluid systems has been challenging because they can be easily disturbed as the specimen is prepared. Although the system can be characterized by indirect methods such as x-ray scattering, chromatography, and conductivity, the analysis can be complicated when the system has more than one type of morphology with a broad size distribution. In contrast, direct methods are intuitive and straightforward, thus it can elucidate the supramolecular structures.[2]

In this work, we studied water/alkenes/nonylphenol ethoxylates surfactant microemulsion processing,[3] processed using dilution and quenching of some microemulsions to make a dilute emulsion which is monodisperse micelles swollen with very insoluble oils (Figure 1). Though the most common way to make a dilute emulsion is with a microfluidizer, it is more convenient to use a phase change emulsification. This low-energy method allows us to create very stable vesicular structures from microemulsions (Figure 2). However, the spontaneous emulsification is not systematically understood why the success of some particular routes depend sensitively on the initial microemulsions and the cosurfactant composition of microemulsions.

Here, we have successfully achieved the formation of using three complementary methods: cryogenic transmission electron microscopy (Cryo-TEM), cryogenic scanning electron microscopy (Cryo-SEM), and freeze-fracture electron-beam evaporated replicate TEM (FF-TEM) to understand physical changes found at every stage of a dilute emulsion development. The complementary use of these techniques shows the behavior of these complex fluid microstructure transitions at multiple scales. Cryo-TEM alone cannot show crucial, large-scale structures, but cryo-SEM can. Cryo-SEM alone cannot show where the oil is, but FF-TEM can. Together, those methods reveal a multilayer vesicle, intermediate that seems reasonable for long-term stability of the “a dilute emulsion.”

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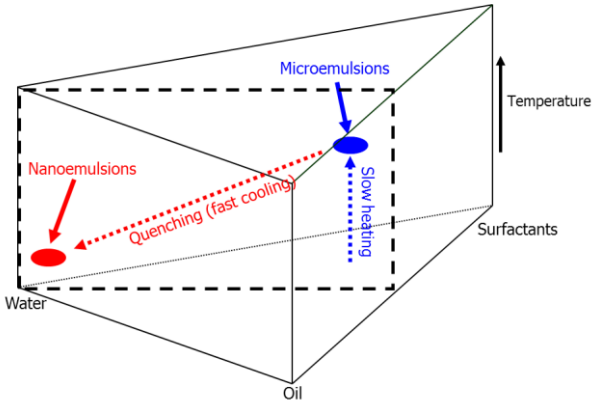

Figure 1. Schematic pathway of a dilute emulsion preparation with a simple phase change emulsification method.

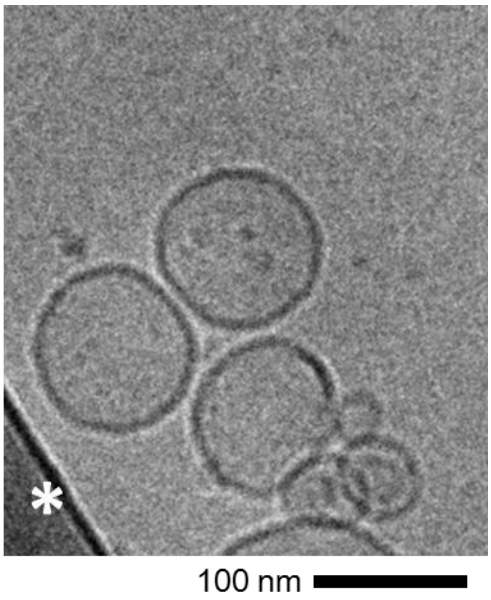

Figure 2. Representative Cryo-EM micrographs of fresh dilute emulsions obtained from a H_2O/*n*-hexadecane/nonylphenol ethoxylates surfactant system. (*) is a lacey carbon strut.

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