CHARACTERIZATION OF EMULSIONS WITH ACOUSTIC SPECTROSCOPY

Emulsions are relatively simple particulate systems that can be characterized by a variety of analytical methods. Acoustic spectroscopy can measure emulsion droplet size at concentrations up to 50 volume %, allowing characterization without dilution. This capability allows measurement of systems that cannot be characterized by other methods or that are sensitive to dilution.

Summary
There are many instances of successful characterization of the particle size distribution and zeta potential of emulsion droplets using acoustic spectroscopy [1, 2]. This application note is a repetition to some extent of McClements' work [3] with hexadecane-in-water and water-in-oil emulsions, to show the range of experiments that can be conducted with acoustic measurements.

Measuring Emulsion Stability
An emulsion was prepared containing 25% by weight of hexadecane in water. The measured attenuation spectra (Figure 1) exhibited a pronounced time dependence. The sound attenuation was found to increase in magnitude as time elapsed. This increase in the attenuation corresponded to the droplet population becoming smaller in size. The median droplet size was reduced by almost two times during a half an hour experiment.

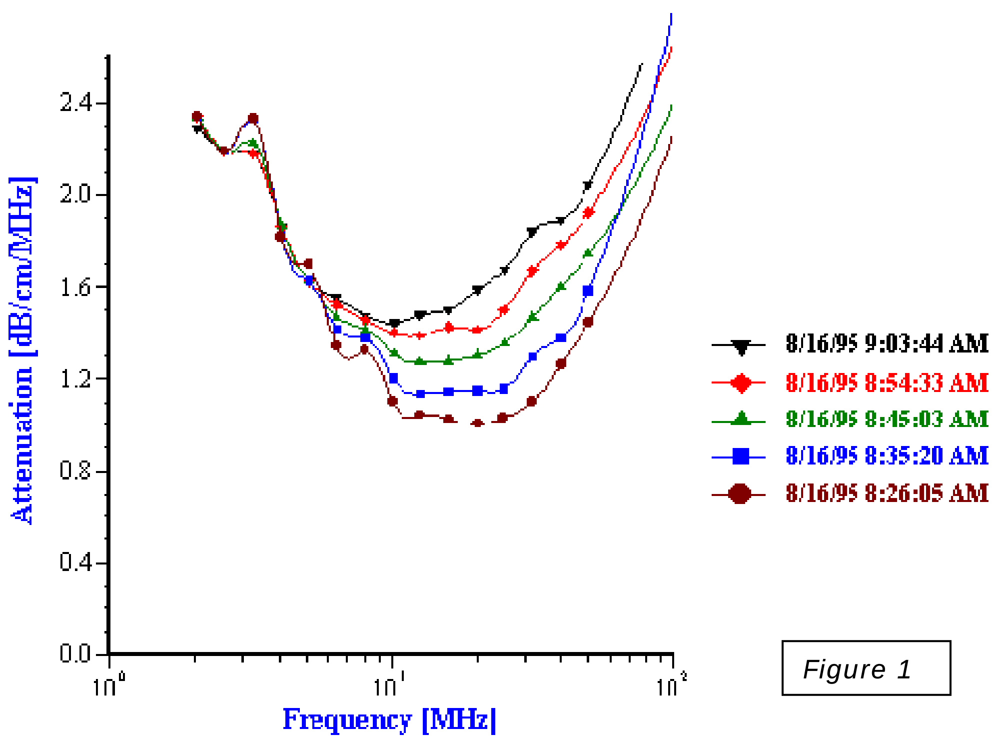

Figure 1

This reduction of the droplet size was caused by the shear induced by a magnetic stirrer used in the sample chamber of the DT-1200 instrument. As the emulsion was stirred, the larger drops were fragmented into smaller droplets. Figure 2 shows the progression of the particle size distribution with time.

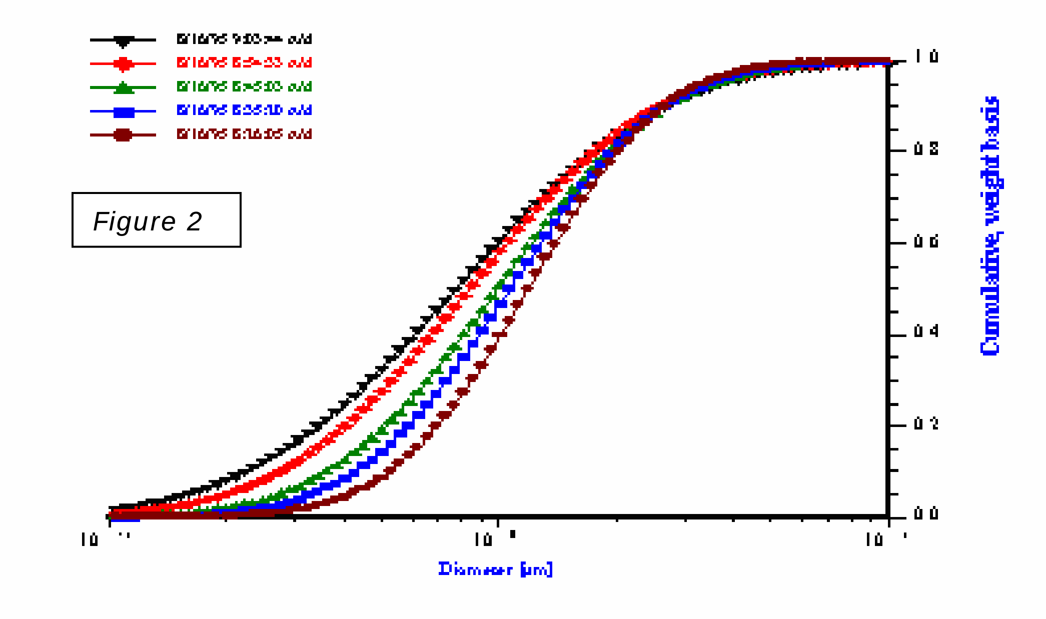

Figure 2

Surfactant Effects on Emulsion Formation

Another important parameter affecting emulsions is the surfactant concentration that affects surface chemistry. This factor was tested for a reverse water-in-oil emulsion. The oil phase was simply commercially available car lubricating oil diluted twice with paint thinner in order to reduce the viscosity of the final sample. Figure 3 illustrates results for emulsions prepared with 6% by weight of water.

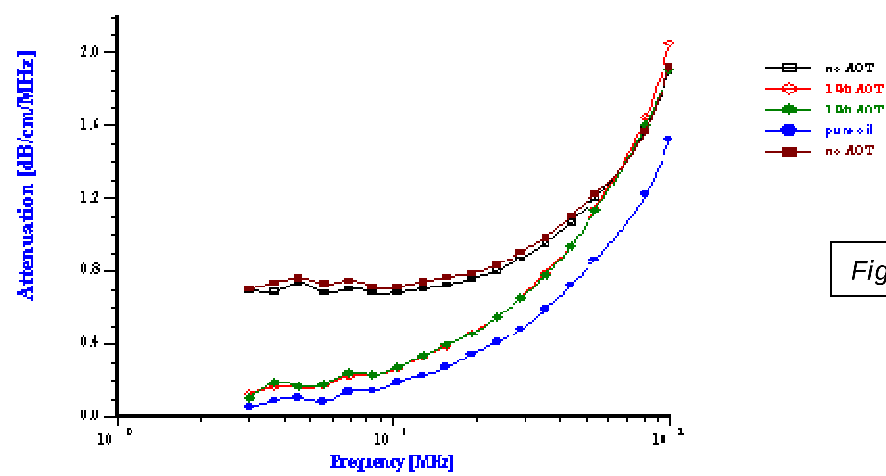

Figure 3

This Figure shows the attenuation spectra for three samples. The first sample was a pure oil phase and exhibited the lowest attenuation.

It is important to measure the attenuation of the pure dispersion medium when a new liquid is evaluated. In this particular case, the intrinsic attenuation of the oil phase was almost 150 dB/cm at 100 MHz which is more than seven times higher than for water. This intrinsic attenuation is a very important contribution to the attenuation of ultrasound in emulsions. It is the background for characterizing emulsion system.

The emulsion without added surfactant was measured twice with two different sample loads. As the water content was increased the attenuation became greater in magnitude. For this system, the attenuation was found to be quite stable with time.

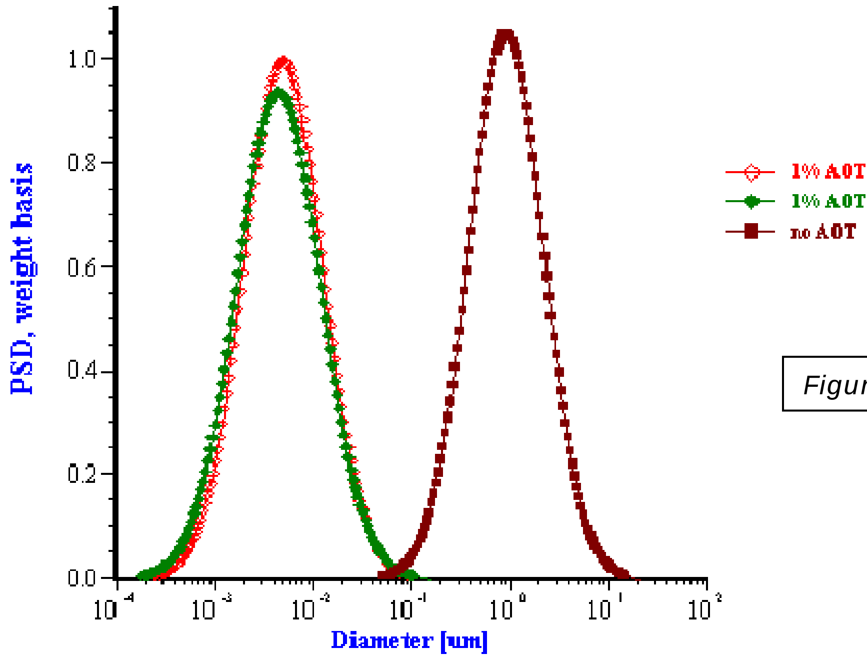

Figure 4

Addition of 1% by weight AOT (sodium bis 2-ethylhexyl sulfosuccinate) changed the attenuation spectra dramatically. This new emulsion with modified surface chemistry was measured two times in order to show reproducibility. The corresponding particle size distribution is shown in Figure 4 and indicates that the AOT converted the regular emulsion into a microemulsion as one could expect.

Conclusion
These experiments analyzed on the HORIBA DT-1200 (figure 5) proved that the acoustic technique is capable of characterizing the particle size distribution of relatively stable emulsions. In many instances emulsions are found that are not stable at the dispersed volume concentration required to obtain sufficient attenuation signals (usually above 0.5 %).

Hazy water in fuel emulsions (diesel, jet fuel, gasoline) may exist at low water concentrations of only a few 100 ppm volume (0.01%) of dispersed water. Attempts at characterizing these systems without added surfactant resulted in unstable attenuation spectra and water droplets were discovered to separate from the bulk emulsion and settle out on the chamber walls.

Figure 5: DT-1200 Analyzer

References:
1. McClements, D.J. “Ultrasonic Characterization of Emulsions and Suspensions”, Adv. in Colloid and Interface Sci., 37 (1991) 33-72
2. Hunter, R.J. “Review. Recent developments in the electroacoustic characterization of colloidal suspensions and emulsions”, Colloids and Surfaces, 141, 37-65 (1998)
3. Dickinson, E., McClements, D.J. and Povey, M.J.W. “Ultrasonic investigation of the particle size dependence of crystallization in n-hexadecane-in-water emulsions”, J.Colloid and Interface Sci., 142,1, 103-110 (1991

For further information on this document or our products, please contact:
Horiba Instruments, Inc.
17671 Armstrong Ave.
Irvine, CA 92614 USA
(949) 250-4811
www.horiba.com