APPLICATION NOTE

Accurate, Rapid Sizing of C_{60} Fullerene Particles

Joshua T. Robinson

Abstract

The explosion in nanotechnology research over the past 10 years has created many opportunities for new and improved materials—such as drugs, paints, alloys, and coatings—but also several challenges in characterizing them accurately. In particular, ultra-small nanoparticles, such as quantum dots and fullerene molecules, are difficult to accurately size in solution. In this application note, we demonstrate how the DelsaMax PRO dynamic light scattering capabilities can accurately size low concentrations of fullerene nanoparticles solubilized in toluene. This application should be of interest to any chemist or material scientist working with nanoparticles in solution.

Introduction

C_{60}, commonly known as fullerene, is an allotrope of carbon discovered in 1985. It has a geodesic dome shape, which inspired the name Buckminsterfullerene, later shortened to fullerene. The structure of C_{60} (Figure 1), comprised of 12 5-carbon rings and 20 6-carbon rings, has an average diameter of 0.7 nm based on theoretical calculations and electron microscopy[1,2]. Because of the extremely small size, accurate measurement of the size of C_{60} in solution has been difficult to impossible for traditional techniques, even at high concentrations. Entirely made up of sp2 carbon, C_{60} is extremely hydrophobic and is only soluble in a few solvents, including toluene[3]. However, with proper surfactant functionalization, C_{60} can be made water-soluble[4]; this has a role in the medicinal applications of C_{60}, as several studies have shown that C_{60} has anti-viral capabilities as well as the ability to act as a photosensitizer for cancer therapy[5]. The most promising application of C_{60}, though, lies in the area of photovoltaic applications. Some of the highest efficiency solar cells to date have been created using a C_{60} derivative [6,6]-phenyl-C_{61}-butyric acid methyl ester coupled with a polythiophene derivative[6].

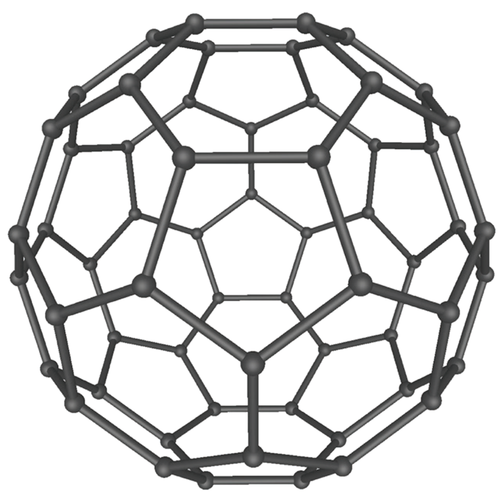

Figure 1. C_{60} structure

Recently, Beckman Coulter, Inc. released a new dynamic light scattering platform, the DelsaMax PRO. With a state-of-the-art Avalanche Photodiode Detector located at 171.5° back-scattering angle (maximizing the signal-to-noise ratio) and strong, 50 mW solid-state diode pumped laser, the weakly-scattering C_{60} particles in toluene solution can be detected, even at ultra-low concentration of 0.04 mg/ml. The DelsaMax software also has customizable limits of analysis for the auto-correlation function, allowing for dust events to be ignored in the analysis; this overcomes a major obstacle, since a single 10 micron speck of dust will scatter light with the same intensity than ~10^{24} C_{60} molecules!

Experimental Section

20 mg of C_{60} molecules (Sigma-Aldrich) were weighed and dissolved in 10 ml of toluene. Gentle bath sonication for 5 minutes was used to fully disperse the C_{60}, resulting in a purple-colored solution. 0.2 ml of this solution was taken and dissolved in 10 ml of toluene, resulting in a final solution concentration of 0.04 mg/ml C_{60}. 100 µl of the 0.04 mg/ml solution was filtered through a 0.1 micron filter (Anatop Syringe filter, Whatman) and directly into the DelsaMax PRO flow cell. This filtration was done quickly to prevent any dissolution of the filter due to exposure to toluene. The measurement was performed at 25° C with an acquisition time of 2 seconds. Based on the model from the optimization calculator in the DelsaMax software, 2,482 acquisitions would be needed to get accurate data; however, the experiment was run with only 500 acquisitions and noise-free, accurate data was obtained, as evidence by the Auto-Correlation Function (Figure 2).

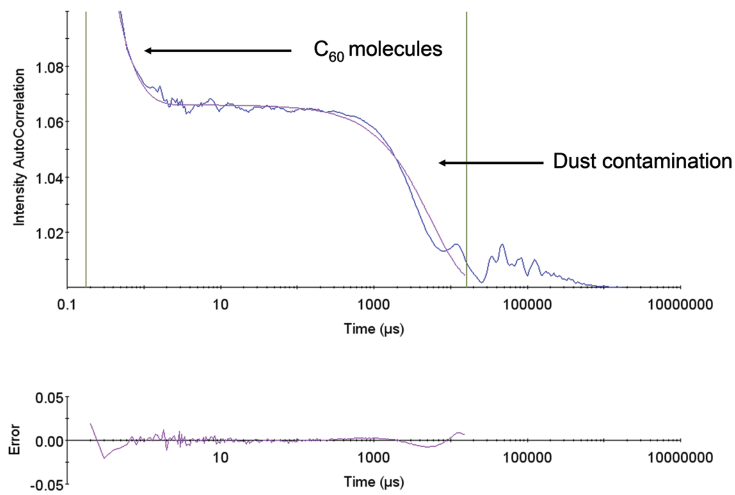

Figure 2. Auto-Correlation Function. The actual data points of the auto-correlation function are in blue, while the regularization fit of the exponential decays is indicated by the purple line. The error from the regularization fit can be seen at the bottom bar of the figure. Note that significant dust contamination, roughly 5-10 micron in size, causes the second dip and subsequent noise in the auto-correlation function. By adjusting the limits of analysis for the auto-correlation function, indicated by the green vertical bars, the effect of dust in the measurement can be minimized.

Results

Using the Regularization method of analysis, which tries to fit the auto-correlation function for multiple species, 100% of the mass in the C_{60} sample was located between 0.6 – 0.8 nm, with a peak diameter of 0.6 nm and a polydispersity of 0.03 nm (Figure 3). The obtained data was within 0.1 nm of the expected value, highlighting the excellent accuracy of the DelsaMax PRO dynamic light scattering instrument, even for small molecules at low concentrations.

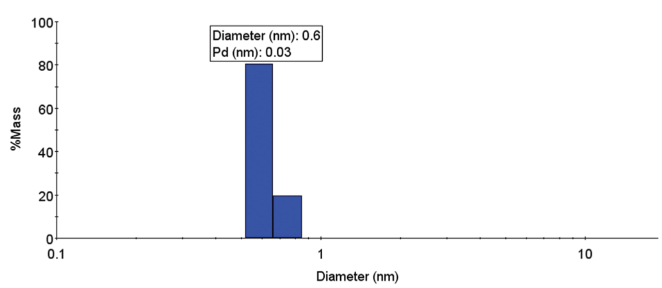

Figure 3. Size Distribution of C_{60} in Toluene. A histogram of the % mass distribution of the C_{60} molecules indicates that 100% of the mass is between 0.6 – 0.8 nm in diameter, in line with the expected results.

References

1. Smalley, Richard E., H. W. Kroto, and J. R. Heath. "C60: Buckminsterfullerene." Nature 318.6042 (1985): 162-163.

2. Quay, C. H. L., et al. "Transport properties of carbon nanotube C_ {60} peapods." Physical Review B 76.7 (2007): 073404.

3. Ruoff, R. S., et al. "Solubility of fullerene (C60) in a variety of solvents." The Journal of Physical Chemistry 97.13 (1993): 3379-3383.

4. Andersson, Thomas, et al. "C60 embedded in γ-cyclodextrin: a water-soluble fullerene." Journal of the Chemical Society, Chemical Communications 8 (1992): 604-606.

5. Bakry, Rania, et al. "Medicinal applications of fullerenes." International journal of nanomedicine 2.4 (2007): 639.

6. Scharber, Markus C., et al. "Design rules for donors in bulk heterojunction solar cells—Towards 10% energy conversion efficiency." Advanced Materials 18.6 (2006): 789-794.

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