Size Separation and Determination of Aqueous C_{60} Colloids by Asymmetric Flow-Field Flow Fractionation (AF4) of with in-Line Dynamic Light Scattering

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Abstract

Current methods for the size determination of nanomaterials in aqueous suspension include dynamic or static light scattering and electron or atomic force microscopy techniques. Light scattering techniques are limited by poor resolution and the scattering intensity dependence on particle size. Microscopy techniques are limited by sample preparation artifacts and are cumbersome when multiple samples must be analyzed. Asymmetric flow-field flow fractionation (AF4) is an open channel size separation technique in which particles are separated by difference in diffusion coefficients. AF4 can operate under an array of solution conditions, including a large range of ionic strengths and surfactants. In addition, AF4 has the unique capability of separating particles ranging from 1 to 500 nm in hydrodynamic diameter (D_h). Presented herein is the first report of the size fractionation of aqueous C_{60} aggregates (aqu/C_{60}) by AF4 coupled in line with dynamic light scattering (DLS) for size determination. Surfactants, which are commonly used to enhance particle stability, were avoided as they may alter particle size. Additionally, to determine the mass of C_{60} in each size range, fractions were collected from the AF4 and the amounts of C_{60} were quantified using LC-APPI-MS. As determined by DLS aqu/C_{60} ranged in size from 80-260 nm in D_h which was verified by the analysis of fractions by DLS in batch mode as well as by TEM. Of the total mass of aqua/C_{60}, 7.7 ± 6.9 % of the aqua/C_{60} mass had D_h less than 80 nm, while 58 ± 32 % had D_h between 80-150 nm and 14 ± 9.2 % of the aqua/C_{60} were between 150-260 nm in D_h. Deposition was observed in the AF4 channel as 79 ± 5.7 % of the initial aqua/C_{60} eluted from the AF4 channel. The use of membranes with hydrophobic and hydrophilic functionalities did not reduce the amount of aqu/C_{60} deposited. As predicted by AF4 theory, detector flow splitting increased the detector response, although increasing the split ratio beyond 80 % of the channel flow resulted in elution of aqu/C_{60} in the split flow stream.