Helping nano-entities discover their nano-identity

Alejandra Castro[1], Mats Leeman[1], J. Ray Runyon[2], Lars Nilsson[1] and Matilda Ulmius Storm[1]

[1]SOLVE Research and Consultancy AB, Lund, Sweden
[2]SOLVE Research and Consultancy LLC, Tucson AZ, US
email: alejandra@solveresearch.com

ABSTRACT

A couple of examples are presented to show the importance of introducing separation techniques as field-flow fractionation (FFF) in the characterization of nanoparticles. Detection of different size populations in a complex sample without sample fractionation is challenging. FFF physically separates the sample according to size before characterization. This allows obtaining information on structure and size across the entire sample distribution from single nanoparticles to large micron-sized aggregates with the help of different on-line spectroscopic and scattering techniques. It is also possible to collect the resulting fractions for additional off-line analysis or to provide monodisperse nanoparticle samples.

INTRODUCTION

Nanoscale materials are becoming increasingly prevalent in our lives. The strategies required to characterize the properties and performance of such materials can be as unique as the material itself.

Compared to conventional batch techniques that directly analyze the size population in a sample, we have experienced that separation of the sample components into narrow size fractions before analysis can provide much more information about the sample. The state-of-the-art technique to do this is field-flow fractionation (FFF). The technique is commercially available, but still not commonly used in the nanomaterial community.

With field-flow fractionation, particles and macromolecules in the size range from 1-1000 nm can be physically separated according to differences in size, and then characterized on-line or off-line with spectroscopic and scattering techniques, such as light absorption, static light scattering or mass spectrometry. Furthermore, the technique allows separation of particles in multiple environments such as high and low pH, different salts and salt concentrations, and conditions that mimic, for instance, the human body.

The gentle conditions used during the fractionation can preserve intact fragile aggregated species. After separation and analysis, it is possible to collect the resulting fractions for additional off-line analysis or to provide monodisperse nanoparticle samples.

EXAMPLES

An iron oxide nanoparticles coated with polyacrylic acid were analyzed for particle size using dynamic light scattering (DLS) (Figure 1) as well as Asymmetrical Flow Field-Flow fractionation (AF4) in combination with multi-angle light scattering and refractive index detection (Figure 12). The commonly used DLS technique demonstrated one population with a broad size distribution, but otherwise no indication of anything remarkable. Using FFF, however, gave a more detailed picture of the size distribution. The analysis detected three size populations, corresponding to nanoparticle cores (as measured with transmission electron microscopy prior to coating), successfully coated particles, as well as aggregated particles. This data illustrates the difficulties involved in detecting different size populations in a complex sample without sample fractionation.

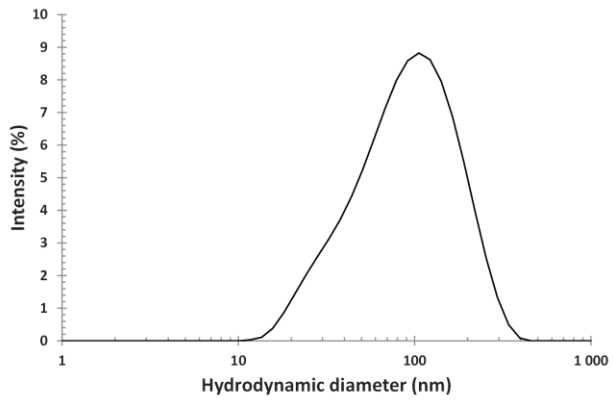

Figure 1. Intensity-based hydrodynamic radius of iron oxide nanoparticles coated with polyacrylic acid analyzed for particle size using dynamic light scattering (DLS).

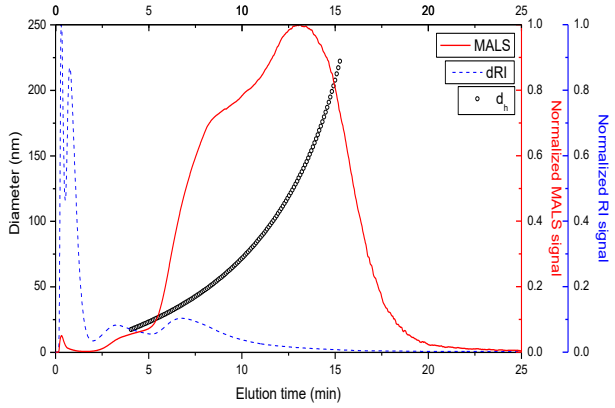

Figure 2. Elution profile of iron oxide nanoparticles coated with polyacrylic acid analyzed by Asymmetrical Flow Field-Flow fractionation (AF4), showing hydrodynamic diameter (circles), normalized MALS signal (red, solid line), and RI signal (blue, dashed line).

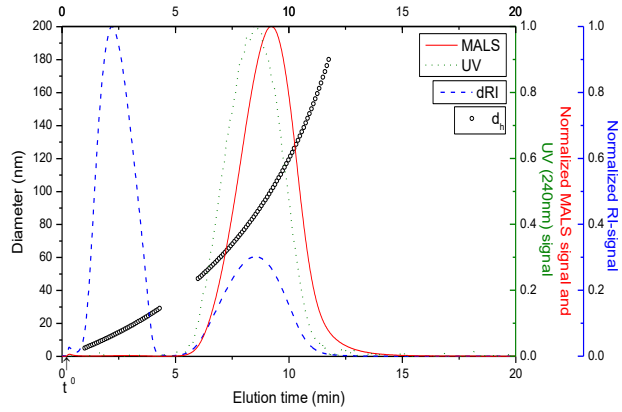

Figure *3*. Elution profile of iron oxide nanoparticle coated with dextran, showing hydrodynamic diameter (circles), normalized MALS signal (red, solid line), UV signal (green, dotted line), and RI signal (blue, dashed line) as a function of elution time. Symbol t^0 refers to the void time.

Characterization with fractionation techniques allows the separation and detection of other population that are not part of the nanoparticles. Figure 3 shows an example from an AF4 analysis of a nanoparticle sample containing two populations. The first population corresponds to free coating material that remains after washing steps. The second population corresponds to the coated nanoparticles. The average diameter of both populations determined by AF4 is 14 and 88 nm, respectively. The data reflects the fact that separation methods like field-flow fractionation do not only determine size but can open the possibility to differentiate the nature of different populations.

CONCLUSIONS

SOLVE Research and Consultancy performs contract analysis and consultation within material characterization for customers from all over the world. Our mission is to provide complete solutions for comprehensive characterization, including customization where we work together to identify and use the most suitable techniques and the flexibility to determine the direction of the study. We provide information on structure, size, molar mass, apparent density etc. across the entire sample distribution from single nanoparticles to large micron-sized aggregates.