Analysis of Iron Oxide Nanoparticles in Organic Solvents for Semiconductor Applications

General Information ID0040

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Introduction

Solvents used in the semiconductor industry can be affected by a variety of contaminants, including metal nanoparticles (NPs). Three of the most commonly used organic solvents in the industry are propylene glycol monomethyl ether acetate (PGMEA), cyclohexanone, and propylene glycol monomethyl ether (PGME) [1]. In this Application Note, we present data on analysis of metal nanoparticles in organic solvents using Asymmetrical Flow Field-Flow Fractionation (AF4) coupled with Multi-Angle Light Scattering (MALS) and Inductively-Coupled Plasma Mass Spectrometry (ICP-MS) detection [2]. Measurement of the NPs' size (i.e. radius of gyration) is also possible using MALS. A schematic for the AF4 channel is shown in Figure 2. The combination of cross flow and channel flow causes size separation over the course of the analysis, with smaller particles eluting to connected detectors before larger particles.

Figure 1: Experimental setup: Postnova AF2000 Asymmetrical Flow Field-Flow Fractionation system (left), Agilent 7900 ICP-MS (right).

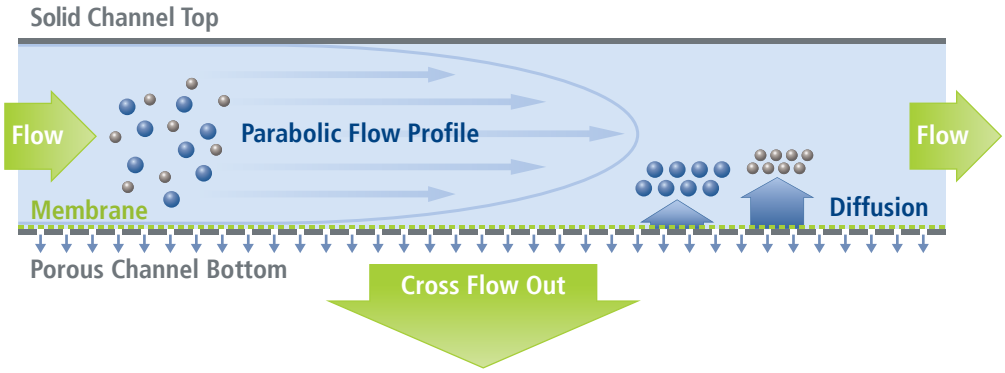

Figure 2: Schematic of the AF4 separation principle.

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Experimental Details and Results

An iron oxide (Fe_2O_3) NP sample, 20 nm average diameter, was used for these experiments with cyclohexanone being both dispersant for the NP sample and mobile phase of the applied fractionation system. Analysis was performed using an AF4 (Postnova AF2000) coupled to a 21-angle MALS (PN3621) and an ICP-MS detector (Agilent 7900), equipped with the Agilent organic solvent sample introduction kit. While MALS enables access to the radius of gyration (Rg) of the NP sample, ICP-MS facilitates its size-resolved elemental identification and quantification. To estimate the AF4-ICP-MS detection limit for Fe_2O_3-NP, three concentrations were analysed (0.5 mg/L, 5 mg/L, and 50 mg/L) and the respective signal-to-noise ratio was calculated.

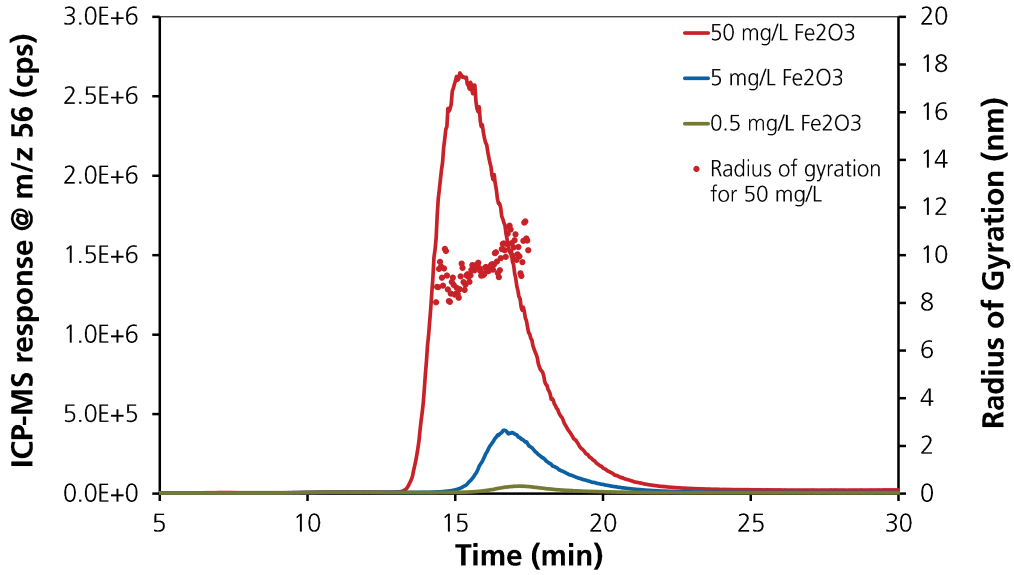

Figure 3: AF4-MALS-ICP-MS fractograms obtained for three different Fe_2O_3-NP concentrations (red line: 50 mg/L, blue line: 5 mg/L, green line: 0.5 mg/L; red dots: radius of gyration) measured in cyclohexanone..

Figure 3 shows the ICP-MS ^{56}Fe isotope response for the size-separated NP sample at three different concentrations. The size distribution is indicated by the Rg data, calculated from the MALS detector, plotted as red dots across the peak for the 50 mg/L concentration. The limit of detection (LOD) by AF4-ICP-MS was calculated to be 6.0 µg/L and the average determined Rg was 9.6 nm, equal to a diameter of 19.2 nm, very close to the NPs' nominal 20 nm diameter.

Conclusion

In this study, we demonstrate the applicability of AF4-MALS-ICP-MS for trace level analysis of inorganic nanoparticles in organic solvents; in particular Fe_2O_3 nanoparticles in cyclohexanone (LOD: 6.0 µg/L). Although this work focused on cyclohexanone, this setup is also compatible with a wide range of further organic solvents, including PGME and PGMEA rendering it a powerful analytical tool for the investigation of particulate contaminants in semiconductor industry.

References

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[2] R. Ruhland, K. Nwoko, M. Perez, J. Feldmann and E.M. Krupp, 2019, Analytical Chemistry, 91, 1, 1164-1170.