Characterization of SiO_2 Nanoparticles by Single Particle - Inductively Coupled Plasma – Tandem Mass Spectroscopy

Diego Pereira Leite[A], Eduardo Bolea-Fernandez[B], Ana Rua-Ibarz [B], Martín Resano[A], Frank Vanhaecke[B], Maite Aramendía[A,C]

[A] Group of Métodos de Análisis Rápidos con Técnicas Espectroscópicas (M.A.R.T.E.) - Department of Analytical Chemistry, Aragón Institute of Engineering Research (I3A), University of Zaragoza, Pedro Cerbuna 12, 50009 Zaragoza, Spain

BDepartment of Analytical Chemistry, Ghent University, Campus Sterre, Krijgslaan 281-S12, 9000 Ghent, Belgium

[C]Centro Universitario de la Defensa de Zaragoza, Academia General Militar de Zaragoza, Carretera de Huesca s/n, 50090 Zaragoza, Spain

Abstract

This work uses the tandem ICP-MS (ICP-MS/MS) for obtaining interference-free conditions to characterize SiO_2 nanoparticles ranging between 80 and 400 nm. These NPs have been detected and accurately characterized. For SiO_2 NPs > 100 nm, it was possible to provide accurate results in a straightforward way, as their signal distributions are well resolved from that of the background.

Introduction

Due to their unique physical and chemical properties, the use of nanomaterials is rapidly growing over the last years. As non-metal oxides, SiO_2 nanoparticles (NPs) are used in a wide variety of applications, such as mechanic polishing or as additives in drugs, food and cosmetics[1]. This massive use of NPs is raising the concern about their potential effects in the environment and on human health, and different international directives exist (e.g. European Commission[2]) urging the need to characterize such materials including information on particle concentration and size of the NPs present in different matrices. In this context, Single Particle Inductively Coupled Plasma – Mass Spectroscopy (SP-ICP-MS) can be considered a suited technique, that provides information as elemental composition, particle size, mass concentration and size distribution[3]. The capabilities to deal with spectral overlaps by the occurrence of strong spectral interferences coming from ubiquitous elements present in the plasma itself. can be enhanced by combining this technique with ICP – Tandem Mass Spectroscopy (ICP-MS/MS). ICP-MS/MS instruments are equipped with two quadrupoles (Q1 and Q2) located before and after an octopole collision/reaction (ORS) cell, thus enabling for a double mass selection. In the MS/MS mode, all ions with different m/z than that of the target nuclide are filtered out by Q1, thus enhancing the control over the collisions/reactions taking place in the ORS and permitting a more efficient resolution of interferences[4].

Experimental

80, 100, 200, 300 and 400 nm NPs suspended in water were obtained from NanoComposix (non-functionalized NanoXact™ Silica, Czech Republic) were properly diluted according to the initial particle concentration size. All measurements were carried out using an Agilent 8800 triple quadrupole ICP-MS/MS instrument (Agilent Technologies, Japan). The instrument is equipped with two quadrupole mass analyzers (Q1 and Q2) and an octopole collision-reaction cell (ORS^3) mounted in-between the two quadrupole units (Q1-ORS-Q2).

Results

The reactivity of Si with H_2 as a reaction gas was evaluated *via* product ion scanning, in which Q1 was set at m/z =28, with Q2 scanning over the entire mass spectrum. The product of this reaction was SiH^+ (m/z =29). However, this gas also react with the polyatomic interferents presents in the the plasma (CO^+ and N_2^+), enabling the analysis free of interferents with the $^{28}Si^+$. Then, SiO_2 were characterized by means of size and particle and mass concentration. Figure 2 shows the frequency distribution for each analyzed NP, where it can be observed an overlap for 80 and 100 nm and a complete separation between the background and NP signal for 200, 300 and 400 nm NPs. Due to this overlap, good recoveries of particle concentration are obtained only for 200 – 400 nm SiO_2 NPs (Table 1). However, good recoveries of mass concentration and particle size can be observed.

Conclusions

The developed method by SP-ICP-MS/MS enables to characterize SiO_2 NPs ranging between 80 and 400 nm. Accurate particle size diameter was achieved for all studied SiO_2 NPs and Good recoveries were obtained for particle and nominal concentrations > 100 nm

References

[1] Montaño, M.D., *et al*, Anal. Chem. 2016, 88, 4733-4711

[2] Commission Recommendation 2011/696/EU, OJ L 275, 20.10.2011

[3] J. Liu, K.E. Murphy, R.I. MacCuspie, M.R. Winchester, Anal. Chem. 2014, 86, 3405-3414

[4] Balcaen, L., *et al*, Analytical Chimica Acta. 2015, 894, 7-19.

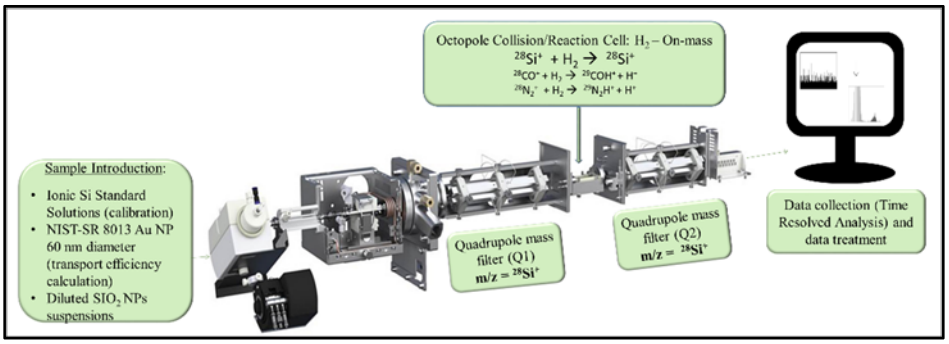

Fig. 1: Instrumental arrangement of SP-ICP-MS/MS

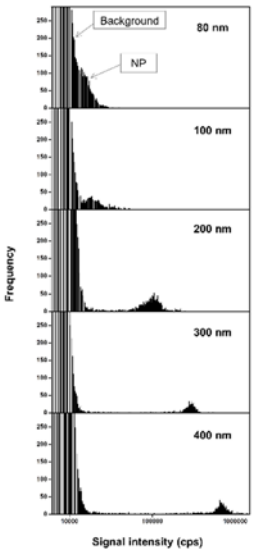

Fig. 2: Frequency distributions of different SiO_2 NP suspensions with different NP diameters when using H_2 as a reaction gas.

Table 1: Characterization of SiO_2 NPs and H_2 (as reaction gases in ICP-MS/MS

Nominal particle size (nm)	Particle diameter (nm)	Particle concentration (x 10^6 particles L^{-1})		Mass concentration (ng/L)	
		Average	Recovery (%)	Average	Recovery (%)
80 nm	90.5 ± 0.8	96 ± 4	64..0 ± 2.9	100 ± 3	99.6 ± 2.7
100 nm	104.4 ± 0.5	63 ± 2	66.3 ± 1.93	104 ± 2	97.6 ± 2.0
200 nm	191.0 ± 0.7	96 ± 3	87.0 ± 3.0	933 ± 33	92.3 ± 3.3
300 nm	284.9 ± 0.5	32 ± 1	93.3 ± 1.9	999 ± 19	96.0 ± 1.8
400 nm	381.2 ±1.9	63 ± 2	89.8 ± 2.2	4944 ± 156	96.0 ± 3.0