

Characterization and Determination of Silver Nanoparticle Using Single Particle-Inductively Coupled Plasma-Mass Spectrometry

YANG Yuan¹, LONG Chen-Lu¹, YANG Zhao-Guang^{1,2}, Li Hai-Pu¹, WANG Qiang^{1,*}

¹ Center for Environment and Water Resources, College of Chemistry and Chemical Engineering, Central South University, Changsha 410083, China

² Shenzhen Graduate School, Central South University, Shenzhen 518057, China

Abstract: A single particle-inductively coupled plasma mass spectrometry (SP-ICP-MS) method was established to detect the size distribution and number concentrations of silver nanoparticles (AgNPs) in dilute aqueous solution. The optimal dwell time was 3 ms to reduce possibility of two or more particles entering simultaneously into detector. An iterative algorithm was applied to distinguish AgNPs as outliers from baseline and dissolved metal ion signal if measured intensity was beyond five time standard deviation of whole data. The size distribution and number concentration of three commercial silver nanoparticle dispersions (nominal diameters of 30, 50 and 100 nm) were determined by SP-ICP-MS. The result of SP-ICP-MS was accurately similar to that of TEM, indicating that SP-ICPMS could be used to detect silver nanoparticles. The particle size detection limit was 25 nm and the limit of number concentration was 8×10^4 particles L^{-1} in dilute solution. Tap water being added with silver nanoparticles was tested to obtain a similar size distribution and number concentration. This method was simple, fast and highly sensitive, and quite feasible to investigate the risk assessment of silver nanoparticles in aqueous environment and monitor silver nanoparticles in drinking water.

Key Words: Silver nanoparticle; Single particle; Inductively coupled plasma-mass spectrometry; Drinking water

1 Introduction

According to the report of Emerging Nanotechnologies Program^[1], there are about 1300 types of commercial nanomaterials introduced to the market and about 20% of them are related to silver nanoscale products. Owing to the special antimicrobial activities, silver nanoparticles (AgNPs) are widely used in fields of textile, water purification instruments, cosmetics, etc. Specific nanoscale materials are not only providing unique characteristic but also increasing toxicity to ecological environment and becoming emerging contaminants^[2,3]. The environmental behavior and ecological risk of AgNPs such as environmental migration, transformation and fate cannot just be predicted by relevant ion or

element. The environmental behavior of AgNPs is more complex than that of the normal pollutant. Now there are not many reports on the determination of AgNPs in water environment. Therefore a qualitative and quantitative method needs to be established urgently to accurately detect AgNPs and study the environmental behavior in water environment.

At present, the most common nanomaterial characterization techniques are dynamic light scattering (DLS), nanotracking analysis (NTA) and electron microscopy (EM). However these methods need high concentration ($mg L^{-1}$) to analyze unknown sample^[4-6], which is not suitable for the analysis of low concentration nanoparticle in water environment. With the advantage of high selectivity, sensitivity and simultaneous multielement determination, inductively coupled plasma mass

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* Corresponding author. Email: qwangesu@163.com

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spectrometry (ICP-MS) is especially suitable for the characterization of these kinds of new materials. Coupled with other techniques, ICP-MS can obtain inorganic nanoparticle size distribution and its character, such as the relationship of freedom ion and nanoparticles. Field flow fractionation (FFF) coupled with ICP-MS was capable of physical separation and analysis of macromolecule, colloid and engineering nanomaterial with 2–1000 nm particle size. However FFF-ICP-MS had poor separation for the determination of Ag^+ , which limited the application to engineering nanomaterials' determination^[7]. Hydrodynamic chromatography (HDC-ICP-MS) is another separation method^[8], but it has a poor resolution compared to that of FFF technology.

Single particle-ICP-MS (SP-ICP-MS) is a high sensitivity analytical method with powerful ability to characterize size distribution of metallic nanoparticle. SP-ICP-MS can save cost because no extra instrument was required. Owing to short analysis time, it is more suitable for the fast determination of metallic nanoparticle in environmental sample. SP-ICPMS collects data with time resolved analysis (TRA) mode and can be used to analyze nanoparticle size distribution and number concentration in aqueous solution. Degueldre *et al.*^[9–11] firstly proposed the theory basis of SP-ICPMS for the determination of colloid particle size distribution in aqueous solution. When the metallic nanoparticle was introduced into the plasma, single nanoparticle generated single strong pulse on short time (< 1 ms). The frequency and the intensity of the pulse signal are related to the number concentration and the mass of the nanoparticle, respectively. Pace *et al.*^[12] elucidated the relationship between nanoparticle intensity and size distribution using transport efficiency and ionic standard solution. The equations are described as follows:

$$f_{\text{NP}} = N_{\text{NP}} Q_{\text{sam}} \eta_n \quad (1)$$

$$W = \eta_n Q_{\text{sam}} t_{\text{dwell}} C \quad (2)$$

$$m_{\text{NP}} = (S_{\text{pulse}} - S_{\text{bgd}}) \eta_n / m f_m \quad (3)$$

$$d_{\text{NP}} = (6m_{\text{NP}} / \rho \pi)^{1/3} \quad (4)$$

where, η_n is transport efficiency, Q_{sam} is sample flow rate (mL min^{-1}), t_{dwell} is dwell time (ms per event), f_{NP} is number of nanoparticle (pulse per event). If the nanoparticle concentration of standard sample is known, η_n can be calculated based on Equation (1). C is analytical ionic element calibration curve equation. According to Equation (2), the mass flux equation W (mass per event) can be established. S_{pulse} is nanoparticle signal, S_{bgd} is background signal. Background signal includes instrument noise and signal from dissolved ions. f_m is the mass fraction of the analyte metal nanoparticles. m is the slope of mass flux equation W . Nanoparticle mass m_{NP} is determined using Equation (3). Assume the analytical nanoparticles are monodisperse and spherical, the diameter of the nanoparticle d_{NP} (nm) can be determined using the nanoparticle mass (Equation (4)), where ρ is the particle density (g cm^{-3}). Nanoparticle number concentration can be calculated basing on Equation (1).

In this study, the size distribution and nanoparticle number concentration of AgNPs in drinking water were determined using SP-ICPMS. By optimizing determination parameters, a quantitative method of monitoring silver nanoparticle in drinking water was established. This method was simple, fast and highly sensitive, and can be used to investigate risk assessment of silver nanoparticle in aqueous environment.

2 Experimental

2.1 Instruments and reagents

Agilent 7700x ICP-MS with MicroMist nebulizer (Agilent, USA) was used for data acquisition. The liquid argon purity was higher than 99.99%. The work parameters of ICP-MS were summed in Table 1.

Data collection and processing software was ICP-MS Mass Hunter Workstation (Agilent, USA) and Excel (Microsoft, USA).

AgNPs suspensions of three different sizes (nominal sizes of 30, 50 and 100 nm) were purchased from NanoComposix (USA). The particles were stabilized with 2 mM citrate. The particle results of TEM and DLS supported by NanoComposix were proved its monodisperse. The TEM diameters of AgNPs were 32.3 nm , $53.4 \text{ nm} \pm 4 \text{ nm}$ and 99.4 nm , respectively. The mass concentration of AgNPs suspension was 0.02 mg mL^{-1} . AgNPs number concentration was $1.1 \times 10^{11} \text{ particles mL}^{-1}$ (32.3 nm), $2.5 \times 10^{10} \text{ particles mL}^{-1}$ ($53.4 \text{ nm} \pm 4 \text{ nm}$) and $3.7 \times 10^9 \text{ particles mL}^{-1}$ (99.4 nm), respectively. Dissolved Ag standards (1000 mg L^{-1} , China Iron & Steel Research Institute, China) for calibration curve was diluted freshly to following: 0, 0.05, 0.2, 0.5 and $2.0 \mu\text{g L}^{-1}$ (0.02% HNO_3 solution). The instrument was tuned daily using tune solution ($1 \mu\text{g L}^{-1}$ Li, Co, Y, Tl, Ce and Ba in 2% (V/V) HNO_3 solution, Agilent, USA).

All AgNPs suspension was diluted in ultrapure water (PERSEE, resistance = $18.2 \text{ M}\Omega \text{ cm}$, China)

All polypropylene volumetric flasks and other vessels were soaked in 20% (V/V) HNO_3 at least 24 h and then washed using ultrapure water.

2.2 Sample preparation and determination

After dilution and before analysis, all samples were placed in ultrasonic water for 15 min to guarantee AgNPs homogeneously in water. All samples were carried out on the day of analysis to avoid AgNPs degradation. The sample nanoparticle

Table 1 Parameters of ICP-MS

Parameters	Value
RF Power (W)	1550
Carrier gas (L min^{-1})	1.05
Spray chamber temperature ($^{\circ}\text{C}$)	2
Nebulizer pump rate (mL min^{-1})	0.36
Data acquisition mode	TRA
Integration time (ms)	3
Acquisition time (s)	60

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