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Multi-dimensional ICP-MS-based methods for a comprehensive assessment of silver physico-chemical speciation and its impact on phytoplankton.

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Silver nanoparticles (AgNPs) are known to rapidly surface-oxidize, leading to ionic silver release responsible for further toxic events. They are thus used in everyday-life products as biocides, potentially occurring at low doses after their release in surface water. The presence of phytoplankton is expected to alter AgNPs fate either by direct interactions or indirectly. The rate of the dissolution process can be thus affected due to the adsorption of released biocomponents at the AgNPs surface, or by their internal biotransformation. This dynamic context, where AgNPs will occur at low doses at bio-interface, challenges the evaluation of their fate, involving changes in surface properties, size-distribution, quantity and effects.

Herein, we evaluated quadrupole based inductively coupled plasma mass spectrometry (ICP-MS) methods with multi-dimensional capabilities for addressing the processes involved in the fate of relatively small 20 nm core-size AgNPs (20nm-AgNPs) when interacting with the model green micro-algae *C. reinhardtii*. The ICP-MS was used in single particle/cell mode (sp/scICP-MS) or associated online with asymmetrical flow field-flow fractionation linked with a fluorescence detector (AF4-FLD-ICP-MS). The stability of AgNPs was addressed after 2h and 24h of dispersion in algal extract of unexposed cells. The silver species formed after 4 hours of internalization by the green algae exposed to 60 ppbAg were characterized, and Ag, P, Mg and Fe cellular distributions obtained were compared to those from ionic silver exposed cells.

Our results show that spICP-MS together with AF4-ICP-MS were performant for quantitively addressed the dissolution rate of 20nm-AgNPs, which involves macromolecular-biocorona formation, as pointed out thanks to AF4-FLD. Despite being identified by spICP-MS, AgNPs mainly resulted in molecular/macromolecular species following their internalization, limiting the quantitative evaluation of nanoparticulate silver by both methods. The cellular intrinsic-metal distributions obtained from scICP-MS measurements weren't affected whereas cells exposed to ionic silver, with similar total content of internalized Ag, massively lost their intrinsic metal contents, suggesting different toxic mode of actions.

All these ICP-MS based methodologies cannot be used as standalone. Their expansion is, however, essential for assessing risk associated with metal-based nanoparticles used as biocides under more environmentally relevant conditions than imposed by other less sensitive techniques.