Sample preparation strategies for food and biological samples prior to nanoparticle detection and imaging

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Accurate and precise characterization of metrics such as size, mass, shape etc. of nanoparticles (NPs) remains a challenging task. In order to determine quantitative metrics that are relevant in food monitoring or in risk assessment, an instrumental separation method like asymmetric field flow fractionation (AFFF, or AF4) coupled on-line to various detectors including static and dynamic light scattering (LS), UV or fluorescence (FL) spectroscopies and ICP-MS have proven useful and powerful [1, 2, 3]. Furthermore, additional information obtained by an imaging method such as transmission electron microscopy (TEM) proved to be necessary for trouble shooting of results obtained from AFFF-LS-ICP-MS.

Aqueous and enzymatic extraction strategies were tested for thorough sample preparation aiming at degrading the sample matrix and to liberate the AgNPs from chicken meat into liquid suspension. The resulting AFFF-ICP-MS fractograms, which corresponded to the enzymatic digests, showed a major nano-peak (about 80 % recovery of AgNPs spiked to the meat) plus new smaller peaks that eluted close to the void volume of the fractograms. Small, but significant shifts in retention time of AFFF peaks were observed for the meat sample extracts and the corresponding neat AgNP suspension, and rendered sizing by way of calibration with AgNPs as sizing standards inaccurate.

In order to gain further insight into the sizes of the separated AgNPs, or their possible dissolved state, fractions of the AFFF eluate were collected and subjected to ICP-MS analysis in single particle (sp) mode. The results showed that the first eluting AFFF peaks contained some dissolved Ag-species and the later eluting peaks primarily contained AgNPs of increasing sizes.

Finally, the possibility of using alkaline pre-treatment of rat spleens prior to sp-ICP-MS analysis of their content of gold nanoparticles (AuNPs) was tested and compared with enzymatic sample preparation [3]. The results showed that the same results, with respect to the obtained number-based size distribution for AuNPs, were obtained for the two preparation methods. In contrast, the alkaline method was by far superior for quantification of AuNPs and was comparable with that obtained by ICP-MS after digestion of the samples in aqua regia. The reason for this is however, not fully understood, and requires further study.

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