**Sample preparation and subsequent analysis of engineered nanomaterials in complex matrices**

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The applicability of a generic scheme [1] to systematically develop methods for detection, characterization, and quantification of engineered nanoparticles (ENPs) in a complex matrix was tested using real products. We selected two sample types and applied the generic sample preparation scheme: 1) a powdered tomato soup which contains the anti-caking agent SiO2 (E551), and 2) a sunscreen which contains TiO2 as UV-filter. Our research focused on sample preparation, aiming to achieve a complete separation of ENP’s from the respective matrix without altering the ENP size distribution and with minimal loss of ENP’s. The generic multi-step sample preparation procedure includes: I) homogenization of the sample; II) ENP separation from the matrix; III) ENP enrichment, and IV) ENP stabilization. The preparation procedure was evaluated by the pre-defined quality criteria which are mass recovery and change in size distribution. The size distribution of the isolated ENPs was determined by asymmetric flow field-flow fractionation (AF4) coupled to multi-angle laser light scattering (MALLS) and inductively-coupled plasma mass spectrometry (ICP-MS) and compared to the size distribution of pristine particles.

We demonstrated that for both sample types the generic sample preparation scheme is valid. For SiO2 in tomato soup, complete matrix removal and Si mass recovery > 90% were achieved using acid digestion supported by heat (90°C) and hydrogen peroxide as oxidation agent. The size distribution of commercially available pristine E551 was comparable with the size distribution of the SiO2 particles isolated from the tomato soup. For TiO2 in sunscreen, TiO2 ENP’s could be isolated by a combination of ultra-centrifugation and hexane washing. Recoveries were sufficiently high to perform FFF-MALLS-ICPMS analysis indicating only a slight shift of the size distribution towards larger diameters. Currently, total TiO2 recoveries are improved by adaptation of ultra-centrifugation and washing steps. In future the developed methodology will be adapted towards the simultaneous extraction of several types of ENP’s (e.g. TiO2, Fe-Oxides).

The proposed generic sample preparation scheme and the applied quality criteria are a very important contribution towards standardized method development in the field of ENP’s in consumer products.

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References

[1] S. Wagner, S. Legros, K. Loeschner, J. Liu, J. Navratilova, R. Grombe, T. P. J. Linsinger, E. H. Larsen, F. von der Kammer and T. Hofmann (2015). First steps towards a generic sample preparation scheme for inorganic engineered nanoparticles in a complex matrix for detection, characterization, and quantification by asymmetric flow-field flow fractionation coupled to multi-angle light scattering and ICP-MS. *J. Anal. At. Spectrom.*, DOI: 10.1039/c4ja00471j