Engineered nanoparticles in cosmetics according to EU 2011/696/EU: sample preparation and analysis

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The applicability of a previously developed generic scheme for detection, characterization, and quantification of engineered nanoparticles (ENPs) in a complex matrix has been extended to real product samples containing unknownparticles. The extended generic multi-step sample preparation procedure includes: 0) pre-characterization of the sample, I) homogenization of the sample, II) ENP separation from the matrix, III) ENP enrichment, and IV) ENP stabilization. In this study, sunscreen, as a common example of complex matrix, which potentially contains TiO as UV-filter was selected to apply the proposed extension of generic sample preparation approach. Pre-characterization in order to identify possibly present ENPs was designed and applied to the sunscreen sample. We demonstrated that the extended generic sample preparation scheme is valid and pre-characterization of the sample is a useful tool to isolate target ENPs for further analysis. TiO ENPs present in the sunscreen could be isolated by a combination of ultra-centrifugation and hexane washing with sufficiently high recoveries for performing further analysis on the particle size. To apply EU 2011/696/EU recommendation where materials are classified as nanomaterials based on number-based particle size distributions; the size distribution of the isolated TiO ENPs was determined by asymmetric flow field-flow fractionation (AF to multi-angle laser light scattering (MALLS) and inductively-coupled plasma-coupled mass spectrometry (ICP-MS). AF -MALLS-ICPMS analysis indicated only a slight shift of the size distribution towards larger diameters. Therefore the mass-based size distribution, which was derived from AF -ICPMS analysis, was converted into a number based size distribution. The applied conversion algorithms are very sensitive towards mass-based signals of small particle sizes. Consequentially high signal noise levels for small particles potentially introduce large errors in the number-based ENP size distribution.

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