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Review

Detection, characterization and quantification of inorganic engineered nanomaterials: A review of techniques and methodological approaches for the analysis of complex samples

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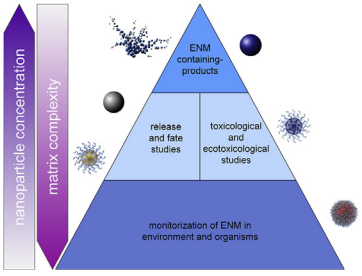
HIGHLIGHTS

- The challenge to analyze inorganic nanomaterials is described.
- Techniques for detection, characterization and quantification of inorganic nanomaterials are presented.
- Sample preparation methods for the analysis of nanomaterials in complex samples are presented.
- Methodological approaches posed by stakeholders for solving nanometrological problems are discussed.

GRAPHICAL ABSTRACT



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ABSTRACT

The increasing demand of analytical information related to inorganic engineered nanomaterials requires the adaptation of existing techniques and methods, or the development of new ones. The challenge for the analytical sciences has been to consider the nanoparticles as a new sort of analytes, involving both chemical (composition, mass and number concentration) and physical information (e.g. size, shape, aggregation). Moreover, information about the species derived from the nanoparticles themselves and their transformations must also be supplied. Whereas techniques commonly used for nanoparticle characterization, such as light scattering techniques, show serious limitations when applied to complex samples, other well-established techniques, like electron microscopy and atomic spectrometry, can provide useful information in most cases. Furthermore, separation techniques, including flow field flow fractionation, capillary electrophoresis and hydrodynamic chromatography, are moving to the nano domain, mostly hyphenated to inductively coupled plasma mass spectrometry as element specific detector. Emerging techniques based on the detection of single nanoparticles by using ICP-MS, but also coulometry, are in their way to gain a position. Chemical sensors selective to nanoparticles are in their early stages, but they are very promising considering their portability and simplicity. Although the field is in continuous evolution, at this moment it is moving from proofs-of-concept in simple matrices to methods dealing with matrices of higher complexity and relevant analyte concentrations. To achieve this goal, sample preparation methods are essential to manage such complex situations. Apart from size fractionation methods, matrix digestion, extraction and concentration methods capable of preserving the nature of the nanoparticles are being developed. This review presents and discusses the state-of-the-art analytical techniques and sample preparation methods suitable for dealing with complex samples.

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Single- and multi-method approaches applied to solve the nanometrological challenges posed by a variety of stakeholders are also presented.

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List of acronyms

AF4	asymmetric flow field-flow fractionation
AGE	agarose gel electrophoresis
CBED	convergent-beam electron diffraction
CE	capillary electrophoresis
CPE	cloud point extraction
DLS	dynamic light scattering
EDS	energy dispersive X-ray spectroscopy
EELS	electron energy-loss spectroscopy
ENM	engineered nanomaterial
ENP	engineered nanoparticle
ESEM	environmental scanning electron microscopy
ET-AAS	electrothermal atomic absorption
EXAFS	extended X-ray absorption fine structure
FESEM	field-emission scanning electron microscopy
FFF	field-flow fractionation
FlFFF	flow field-flow fractionation
GE	gel electrophoresis
HDC	hydrodynamic chromatography
ICP-MS	inductively coupled plasma mass spectrometry
ICP-OES	inductively coupled plasma optical emission spectrometry
MALS	multi-angle light-scattering
MWCO	molecular-weight cutoff
NOM	natural organic matter
NP	nanoparticle
NPM	natural particulate matter
NTA	nanoparticle tracking analysis
PAGE	polyacrylamide gel electrophoresis
PCC	particle collision coulometry
SAED	selected-area electron diffraction
SdFFF	sedimentation field-flow fractionation
SDS	sodium dodecyl sulfate
SEC	size exclusion chromatography
SEM	scanning electron microscopy
SPE	solid phase extraction
SP-ICP-MS	single particle ICP-MS
TEM	transmission electron microscopy
TMAH	tetramethylammonium hydroxide
UF	ultrafiltration
VIP	voltammetry of immobilized particles
XANES	X-ray absorption near edge structure
XAS	X-ray absorption spectroscopy