



Current limitations and challenges in nanowaste detection, characterisation and monitoring



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ABSTRACT

Engineered nanomaterials (ENMs) are already extensively used in diverse consumer products. Along the life cycle of a nano-enabled product, ENMs can be released and subsequently accumulate in the environment. Material flow models also indicate that a variety of ENMs may accumulate in waste streams. Therefore, a new type of waste, so-called nanowaste, is generated when end-of-life ENMs and nano-enabled products are disposed of. In terms of the precautionary principle, environmental monitoring of end-of-life ENMs is crucial to allow assessment of the potential impact of nanowaste on our ecosystem. Trace analysis and quantification of nanoparticulate species is very challenging because of the variety of ENM types that are used in products and low concentrations of nanowaste expected in complex environmental media. In the framework of this paper, challenges in nanowaste characterisation and appropriate analytical techniques which can be applied to nanowaste analysis are summarised. Recent case studies focussing on the characterisation of ENMs in waste streams are discussed. Most studies aim to investigate the fate of nanowaste during incineration, particularly considering aerosol measurements; whereas, detailed studies focusing on the potential release of nanowaste during waste recycling processes are currently not available. In terms of suitable analytical methods, separation techniques coupled to spectrometry-based methods are promising tools to detect nanowaste and determine particle size distribution in liquid waste samples. Standardised leaching protocols can be applied to generate soluble fractions stemming from solid wastes, while micro- and ultrafiltration can be used to enrich nanoparticulate species. Imaging techniques combined with X-ray-based methods are powerful tools for determining particle size, morphology and screening elemental composition. However, quantification of nanowaste is currently hampered due to the problem to differentiate engineered from naturally-occurring nanoparticles. A promising approach to face these challenges in nanowaste characterisation might be the application of nanotracers with unique optical properties, elemental or isotopic fingerprints. At present, there is also a need to develop and standardise analytical protocols regarding nanowaste sampling, separation and quantification. In general, more experimental studies are needed to examine the fate and transport of ENMs in waste streams and to deduce transfer coefficients, respectively to develop reliable material flow models.

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1. Introduction

Nanoscale particles and colloids have always been part of our environment. Such particles are in the size range of 1–100 nm and can originate from natural sources (e.g. as aerosols caused by forest fires or volcanic activities), but manufactured nanoscale particles now also have increasing significance in the environment.

This stems from their obvious commercial interest for a wide range of applications primarily due to their nano-specific and tuneable physicochemical properties. As a result, there are both naturally-occurring and engineered nanomaterials (“NNMs” and “ENMs”) present in our environment and in waste streams. The recommendation of the European Commission (EC, 2011) addresses also both types: “a nanomaterial means a natural, incidental or manufactured material containing particles, in an unbound state or as an aggregate or as an agglomerate and where, for 50% or more of the particles in the number size distribution, one or more external dimensions are in the size range 1 nm – 100 nm.”

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ENMs have become a significant topic of research due to their potential toxicity and risks that may arise during their manufacturing, application, use and disposal (EC, 2012; Savolainen et al., 2014). Commonly used ENMs such as nano-TiO₂ and -SiO₂ are already produced in large volumes; approximately 3000 tons of nano-TiO₂ and 5500 tons of nano-SiO₂ are produced worldwide per year (Piccinno et al., 2012). In the framework of the Project on Emerging Nanotechnologies (last updated in 2013) more than 1600 consumer products were listed in a database to be containing ENMs (PEN, 2014), while *The Nanodatabase* (DTU Environment, 2015) currently lists more than 1400 products. The database of NANOWERK (2015) catalogues more than 2600 different types of commercial ENMs indicating that there is already a wide range of uses of ENMs in the processing industry (e.g. as additives or catalysts) and in consumer products – various applications are summarised in detail in recent publications (e.g., EC, 2012; Marcoux et al., 2013; Yang and Westerhoff, 2014). Ultimately, products containing such nanomaterials reach their end-of-life and the majority of ENMs will therefore end up and may subsequently accumulate in waste streams (Caballero-Guzman et al., 2015; Gottschalk et al., 2013; Keller and Lazareva, 2013; Mueller and Nowack, 2008; Sun et al., 2014).

Therefore, implementation of suitable analytical methods is crucial to assess potential release pathways of ENMs, both during waste treatment processes and from “final sinks” (e.g. landfills), and to create and validate model parameters such as transfer coefficients that are needed for modelling the fate of ENMs along a product’s life cycle. In order to monitor and assess the fate and behaviour of ENMs in the environment, currently available analytical techniques have to be adapted with respect to the nanoparticle type of interest (e.g., in-/organic, un-/coated, hydrophobic/-philic, colloidal un-/stable?), detection and quantification at low concentrations and differentiation from complex background (Hasselov et al., 2008; Howard, 2010; Kammer et al., 2012; Schaumann et al., 2014; Tiede et al., 2008).

The present contribution aims to provide a broad overview on available analytical techniques that are currently applicable for detection and characterisation of ENMs, and the reader will be guided to other selected papers focusing on specific analytical methods. Some analytical techniques are discussed regarding their applicability for the challenging analysis of complex and heterogeneous waste matrices, and case studies that provide examples of current approaches for characterisation and detection of nanowaste are reviewed. In terms of environmental protection as well as occupational health and safety aspects, all dispersion media are considered because end-of-life ENMs are likely to occur in diverse waste streams (Gottschalk et al., 2013; Keller and Lazareva, 2014; Sun et al., 2014), and may be present in the solid, liquid and also gaseous phase.

2. Current challenges in nanowaste management

In general, ENMs can be released at any point in the life cycle of a product: during production, use phase, or at the end-of-life stage of products via mechanical, thermal and chemical processes such as abrasion, combustion, corrosion and leaching (Gottschalk et al., 2009; Mueller and Nowack, 2008; Nowack et al., 2013; Petersen et al., 2011). Studies on the use of nano-Ag, -SiO₂ or contained in paints as additives indicate that very small proportions of ENMs are released during the usage phase and, subsequently, ENMs remain largely in the product’s matrix (Al-Kattan et al., 2014, 2013; Kaegi et al., 2010, 2008). Currently, very little is known on the potential release of ENMs during the end-of-life phase. Realistic mechanisms for ENM release along the product’s life cycle, such as machining, washing, weathering, incineration, and

release through contact with humans (e.g. sweating), are summarised by Froggett et al. (2014). The authors reviewed some experiments showing that also nanoscale debris can be released from composite materials, regardless of whether ENMs are originally present or absent in a sample. This example highlights one of the challenges of generating realistic models as distinguishing between this nanoscale debris from ENM release stemming from nano-enabled products can be extremely difficult from an analytical perspective.

In terms of waste management, Boldrin et al. (2014) as well as Marcoux et al. (2013) state that nanowaste can only be generated in the presence of ENMs, when unused ENMs or contaminated items are directly disposed of during production processes or when ENM containing products reach their end-of-life. Currently, very little is known about the transfer of ENMs from solid waste matrices to the liquid phase like in landfill leachates (Reinhart et al., 2010), during incineration processes (Bouillard et al., 2013; Price et al., 2014), or during recycling processes where ENMs can become airborne. Therefore, approaches and analytical methods are needed to detect ENMs in solid, liquid and gaseous waste samples in order to address these knowledge gaps.

Of paramount importance in nanowaste detection and tracking ENMs in complex waste matrices is a sound knowledge of their chemical composition. 61 different chemical elements are known to be used in commercially available ENMs (see Fig. 1). These elements were identified based on market research and on the working paper of the European Commission (EC, 2012). In order to examine the fate of ENMs in waste streams, obtaining detailed chemical information at the point-of-manufacturing of ENMs (i.e., element profiles or fingerprints) is a particularly valuable approach to facilitate later comparison from samples in waste streams. Such matrices are, by their nature, very complex and therefore methodological aspects such as sampling and detection must be adapted to cope with this. Thus, Walser and Gottschalk (2014) suggest that the use of ENM fingerprints based on distinctive abundances of rare earth metals in source material or isotope labelling can be used in order to be able to differentiate ENMs from nanoparticulate emissions or natural background sources of the constitutional elements. For example, Kammer et al. (2012) proposed the use of Ce:Nd ratios to discriminate synthetic CeO₂ from their natural counterparts, while Gondikas et al. (2014) determined elemental ratios of Ti with many other rare-earth elements to distinguish nano-TiO₂ released from sunscreens from naturally-occurring TiO₂ nanoparticles. Grass et al. (2014) even used DNA barcodes to label silica particles in order to trace them in waste water treatment plants. Experiments from Schierz et al. (2012) or Petersen et al. (2008) show the traceability of radiolabelled single wall carbon nanotubes (¹⁴C-SWNT). Part et al. (2015) made use of the distinctive fluorescent properties of semiconducting quantum dots in order to trace these surface modified CdTe nanoparticles in landfill leachates.

Wagner et al. (2014) pointed out that a major difference of ENMs to NNMs is that ENMs are often surface modified (e.g., by organic coatings or artificial surfactants) in order to increase their colloidal stability aiming to increase the processability in nano-enabled products. In general, surface properties, mobility and dissolution behaviour predominantly determine the fate and transport of ENMs in the environment (Wagner et al., 2014). Currently, very little is known about potential alteration or degradation of organic ENM surface coatings via chemical, physical or biological processes during waste treatment processes. It is also noted that possible transformation and alteration processes can also affect bare (uncoated) ENMs. Such processes may occur along the entire life cycle of nano-enabled products and are summarised by Mitrano et al. (2015) and Ramakrishnan et al. (2015). In terms of risk assessment and nanowaste management, alteration and

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