



Detection of nanoparticles in Dutch surface waters

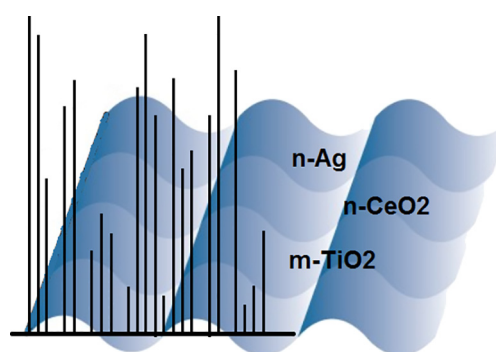
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HIGHLIGHTS

- An analytical method has been validated for the measurement of nanoparticles at low environmental concentrations.
- The study confirms the presence of nano-sized Ag and CeO₂ particles and micro-sized TiO₂ particles in surface waters.
- Particle concentrations range from 0.8 ng/L for Ag to 3.1 µg/L for TiO₂.
- The results of the study confirm the predicted environmental concentrations.

GRAPHICAL ABSTRACT



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ABSTRACT

Nano-enabled consumer products are a likely source of nanoparticles in the environment and a number of studies have shown the release of nanoparticles from commercial products. Predicted environmental concentrations have been calculated but there is a need for real measurement data to validate these calculations. However, the detection of engineered nanoparticles in environmental matrices is challenging because of the low predicted environmental concentrations which may be in the ng/L range. In this study nanosized Ag, CeO₂ and TiO₂ have been measured in multiple surface water samples collected along the rivers Meuse and IJssel in the Netherlands using single-particle ICP-MS as measurement technique. Validation of the analytical method showed its capability to quantitatively determine nanoparticles at low concentrations. Concentration mass detection limits for Ag, CeO₂ and TiO₂ were 0.1 ng/L, 0.05 ng/L and 10 ng/L respectively. Size detection limits for Ag, CeO₂ and TiO₂ were 14, 10 and 100 nm. The results of the study confirm the presence of nano-sized Ag and CeO₂ particles and micro-sized TiO₂ particles in these surface waters. n-Ag was present in all samples in concentrations ranging from 0.3 to 2.5 ng/L with an average concentration of 0.8 ng/L and an average particle size of 15 nm. n-CeO₂ was found in all samples with concentrations ranging from 0.4 to 5.2 ng/L with an average concentration of 2.7 ng/L and an average particle size of 19 nm. Finally, µ-TiO₂ was found in all samples with a concentration ranging from 0.2 to 8.1 µg/L with an average concentration of 3.1 µg/L and an average particle size of 300 nm. The particle sizes that were found are comparable with the particle sizes that are used in nanomaterial applications and consumer products. The nanoparticle concentrations confirm the predicted environmental concentrations values in water for all three nanoparticles.

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1. Introduction

Engineered nanomaterials (ENMs) are promising for applications in industry and consumer products. Like other chemicals that are manufactured and used in products, ENMs may be released to the environment (air, water, soil, sediment and biota) during the production, use, and disposal phases of these products. The rate at which this takes place depends on the quantity of ENMs in the product, how robustly they were incorporated, the lifetime and the properties of the product. For example, >75% of ENMs used in cosmetics are estimated to be released during use (Keller et al., 2014). It is difficult to find reliable and recent production data for ENMs, however, a survey of 2012 showed that the most produced ENM is TiO₂ with production volumes up to 10,000 t worldwide (Piccinno et al., 2012). CeO₂, FeO_x, AlO_x, ZnO and carbon nanotubes (CNT) have production volumes in the range of 100 to 1000 t/year while the production data for SiO₂ covered the whole range, from <10 to >10,000 t/year. Ag is produced only in moderate quantities estimated to be 100 t/year worldwide and 10 t/year for Europe (Piccinno et al., 2012).

Environmental release of ENMs during the product life cycle potentially has far-reaching impacts upon natural ecosystems and ultimately human health. Traces of anthropogenic contamination with persistent chemicals like dioxins, PCB's, brominated flame retardants, poly-fluorinated compounds and heavy metals can now be found in all compartments of the environment, in the human food production chain, and in most individuals (Peters et al., 2008). Although nanomaterials may be not as persistent as these global contaminants, they may very well become a new type of environmental contaminant. Currently there is little or no analytical data on the environmental occurrence of ENM. Predicted environmental concentrations (PEC) for ENM range from 0.00004 to 0.619 µg/L for n-Ag, from <0.0001 to 0.1 µg/L for n-CeO₂ and from 0.0002 to 24.5 µg/L for n-TiO₂ (see Table 1).

The potential environmental releases of ENMs from production through use to release or disposal are shown in Fig. 1. These include point source emissions, such as those from industrial installations or from urban waste water treatment plants, and diffuse emissions, i.e. emissions from products along their life cycle. The quantity of ENMs released during product use depends on the quantity incorporated into the product, how robustly they were incorporated into the product, the lifetime of the product, and the nature of the product's use (Benn and Westerhoff, 2008). ENMs in consumer products such as fabrics, personal care products and food (including Ag, TiO₂ and SiO₂) have a higher likelihood of entering the environment because they can be washed down drains during household use. For example, the majority of ENMs

used in cosmetics, such as TiO₂, silica (SiO₂) and aluminium oxide (Al₂O₃), are estimated to be released during use. Thus, expected releases of ENMs are associated with products that have high release rates and/or high use rates, and these diffuse emissions likely form the majority of the total emissions.

Although the majority of ENMs are likely to be removed through conventional biological wastewater treatment, ENMs have been detected in wastewater effluents at concentrations in the range of <12 ng/L⁵ for Ag nanoparticles and <2 to 20 µg/L for TiO₂ (Westerhoff et al., 2011). Wastewater treatment plant bio-solids are often used as a fertilizer on agricultural land. ENMs present in land-applied bio-solids could be transported to surface waters with other fine particulate matter in runoff water (Mahdi et al., 2017). In addition to agricultural runoff, ENMs may also enter surface waters from urban runoff, including storm water, which may contain ENMs from products such as exterior paints and coatings (Kaegi et al., 2008; Kaegi et al., 2010) or from aerial deposition or spills of fuel additives such as CeO₂ (Johnson and Park, 2012).

Pristine ENMs are generally chemically well defined, however exposing them to the environment may result in an altered state that may be different from their initial ENM form. This has been shown to be largely due to the presence of natural organic matter (NOM, e.g. humic and fulvic acids), that is present in concentrations typically several orders of magnitude higher than the ENMs ranging from 1 to 20 mg/L in freshwaters (Kritzbeg and Ekström, 2012). Dissolution and oxidation-reduction reactions can alter the original structure of the ENM. Particle coatings may be removed or replaced by different coatings such as polymeric-like molecules. NOM substances in particular have been shown to overcoat or replace the surface groups of ENMs. Proteins and other small organic molecules have been known to interact with ENM surfaces resulting in changes in the aggregation state of the ENMs. Hetero-aggregation with natural colloids is therefore likely to control the fate of most ENM (Barton et al., 2015; De Klein et al., 2016; Guo et al., 2016; Labille et al., 2015; Wang et al., 2015; Wilkinson et al., 1997).

However, our limited knowledge of the interaction of nanomaterials with natural organic matter is probably the biggest limitation in exposure modelling (Arvidsson et al., 2011; Garner et al., 2017). As a result, there is an urgent need for data to validate the model predictions. Recently, a number of papers were published describing the determination of organic and inorganic nanoparticles in environmental samples. However, these papers focused on analytical techniques, reviewed earlier results (mostly in waste water), or gave a very broad overview of all environmental matrices (Bauerlein et al., 2017; Laborda et al.,

Table 1

A summary of predicted environmental concentrations (PEC) in surface water of n-Ag, n-CeO₂ and n-TiO₂ reported in the literature.

n-Ag µg/L	n-CeO ₂ µg/L	n-TiO ₂ µg/L	Method	Matrix	Year and reference
0.03–0.08		0.7–16	Model	Surface water	2008 (Mueller and Nowack, 2008)
0.0006–0.0022		0.012–0.057	Model	Surface water EU	2009 (Gottschalk et al., 2009)
0.0001–0.0004		0.002–0.010	Model	Surface water US	2010 (Gottschalk et al., 2009)
0.0006–0.0026		0.016–0.085	Model	Surface water CH	2010 (Gottschalk et al., 2009)
0.029	0.024	1.45	Model	Surface water	2010 (O'Brien and Cummins, 2010)
0.0028–0.619		0.0027–0.27	Model	Surface water	2010 (Musee, 2010)
<0.0001–0.003			Model	Surface water	2011 (Johnson et al., 2011a)
		8.8	Model	Surface water	2011 (Johnson et al., 2011b)
0.00004–0.007		0.01–1.6	Model	Surface water	2011 (Gottschalk et al., 2011)
		0.55–6.48	Analytical	Surface water	2011 (Neal et al., 2011)
0.010–0.03	<0.0001	0.7–24.5	Model	Surface water	2011 (Ferreira da Silva et al., 2011)
0.0005–0.0009		0.4–1.4	Model	Surface water EU	2014 (Sun et al., 2014)
0.0004–0.0007		0.54–3.0	Model	Surface water CH	2014 (Sun et al., 2014)
0.3			Model	Surface water	2015 (Dumont et al., 2015)
<0.0001–0.044	0.0006–0.1	0.0006–0.1	Model	Surface water	2015 (Gottschalk et al., 2015)
<0.0001–0.5		0.0002–5	Model	Surface water	2016 (Good et al., 2016)
<0.1		2.2	Analytical	Surface water	2016 (Donovan et al., 2016)
0.0004–0.0028		0.19–4.4	Model	Surface water	2016 (Sun et al., 2016)

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