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Q3 Application of nanoparticle tracking analysis for 2 characterising the fate of engineered nanoparticles 3 in sediment-water systems

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A B S T R A C T

Novel applications of nanotechnology may lead to the release of engineered nanoparticles (ENPs), which result in concerns over their potential environmental hazardous impact. It is essential for the research workers to be able to quantitatively characterise ENPs in the environment and subsequently to assist the risk assessment of the ENPs. This study hence explored the application of nanoparticle tracking system (NTA) to quantitatively describe the behaviour of the ENPs in natural sediment-water systems. The NTA allows the measurement of both particle number concentration (PNC) and particle size distribution (PSD) of the ENPs. The developed NTA method was applied to a range of gold and magnetite ENPs with a selection of surface properties. The results showed that the positively-charged ENPs interacted more strongly with the sediment than neutral and negatively-charged ENPs. It was also found that the citrate coated Au ENPs had a higher distribution percentage (53%) than 11-mercaptoundecanoic acid coated Au ENPs (20%) and citrate coated magnetite ENPs (21%). The principles of the electrostatic interactions between hard (and soft) acids and bases (HSAB) are used to explain such behaviours; the hard base coating (i.e. citrate ions) will interact more strongly with hard acid (i.e. magnetite) than soft acid (i.e. gold). The results indicate that NTA is a complementary method to existing approaches to characterise the fate and behaviour of ENPs in natural sediment.

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52 Introduction

53 The application and manufacturing of engineered nanoparticles (ENPs) has increased dramatically in the last decades due to their extraordinary properties (Nowack and Bucheli, 2007; Tiede et al., 2009). Consequently, it is inevitable that ENPs will

54 be released into the natural environment (Boxall et al., 2007). Due to the unique properties of ENPs (Tiede et al., 2010), existing methods and models that are used to assess the environmental risk of “traditional” chemicals may not be appropriate for ENPs (Ashby et al., 2009). Based on existing information, it is suggested that, ENPs, like other chemical

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contaminants, may cause irreversible changes following contact with natural environment (Dror et al., 2015). There is therefore an urgent need to develop testing guidelines specific for ENPs (Domingos et al., 2009; Menard et al., 2011; Weinberg et al., 2011). Numerous studies have recently explored the fate and effects of ENPs in surface waters and soils (Bakshi et al., 2015; Bertsch et al., 2012; Bin et al., 2011; Chen et al., 2008, 2010; Dror et al., 2015; Garner and Keller, 2014; Hotze et al., 2010; Ju-Nam and Lead, 2008; Pan and Xing, 2012; Petosa et al., 2010; Quik et al., 2011; Yang et al., 2014; Zhang, 2014). However, limited work has been done on the quantitative fate and behaviour of ENPs in sediment-water systems (Aschberger et al., 2011; Gao et al., 2011; Luo et al., 2011; Peralta-Videoa et al., 2011).

An assessment of the fate and distribution of ENPs in sediment will be critical in the assessment of the potential impacts of ENPs on the natural environment. Sediment is the environmental compartment where most particles will ultimately end up due to aggregation and deposition processes (Nickel et al., 2015). Benthic organisms, which play a key role in the functioning of ecosystems, therefore have the potential to be exposed to higher mass concentrations of an ENP compared to organisms dwelling in the water column (Garric and Thybaud, 2011).

For "traditional" chemicals the distribution in sediment-water systems is usually assessed using batch sorption studies or sediment-water simulation studies such as those described by the OECD 106 and 308 using conventional analytical methods such as gas chromatography-mass spectrometer (GC-MS), liquid chromatograph-mass spectrometer (LC-MS), liquid scintillation counting (LSC) for organic compounds and inductively coupled plasma-mass spectrometry (ICP-MS) or atomic absorption spectrophotometer (AAS) for metals (106, 2000; 308, 2002). These analytical methods define the contaminant mass concentration in the aqueous and solid phases being studied.

However these analytical techniques do not provide important information on the ENPs, such as particle number concentration (PNC) and the particle size distribution (PSD), which may play a critical role in their ecotoxicological effects (Botta et al., 2011). A range of approaches could potentially be used in fate studies to provide information on the PNC and/or PSD including dynamic light scattering (DLS), electrophoretic light scattering (ELS), tunable resistive pulse sensor (TRPS), differential centrifugal sedimentation (DCS), scanning mobility particle sizing (SMPS), hydrodynamic chromatography-ICP-MS (HDC-ICP-MS), field flow fractionation-ICP-MS (FFF-ICP-MS), single particle ICP-MS (SP-ICP-MS) and imaging methods such as scanning electron microscopy (SEM) and transmission electron microscopy (TEM) (Dudkiewicz et al., 2011; Nickel et al., 2015; Tiede et al., 2008). It is well known that techniques such as light scattering, optical microscopy and electron microscopy are limited because they either measure only an averaged sample size distribution, lack the resolution, or are too time consuming to be conveniently used to measure polydispersed ENPs in sediment (Anderson et al., 2013; Bell et al., 2012). These factors can result in ENPs size misinterpretation and subsequently mislead its effect on the fate of ENPs in sediment. The ecotoxicity of ENPs on the sediment-dwelling benthic organisms was also not reliable. For example,

Nickel et al. (2015) was trying to use the OECD 106 to predict the mobility of TiO₂ ENPs in soils and found that no valid DLS or ELS measurements in the pore water or the eluate could be conducted. This is because the DLS failed to accurately measure the size of the polydispersed sample. Anderson et al. (2013) confirmed that DLS measurements made no indication of multimodal sample (e.g. 220, 330, 410 nm). It is recommended that DLS only be used for a priori known mono-dispersed sample. TRPS and DCS provided the greatest resolution and accuracy, being the only techniques to distinguish the polydispersed sample with the sub-populations of the 220, 330, and 410 nm (Anderson et al., 2013). However, the concentration measurement using CPS is reluctant since it measures the light intensity of ENPs and then recalculate into the concentration.

For fate studies, ideally methods would be inexpensive, quick to perform and can reliably measure the PSD and PNC in liquid. One of the methods that meets these criteria is nanoparticle tracking analysis (NTA), a laser-scattering technique, which tracks the Brownian motion of small particles and uses this information to calculate the size of particles (Liu et al., 2012b; Luo et al., 2013). So far NTA has been used to assess the behaviour of ENPs in aqueous environments for environmental, biological, food, and aquatic ecotoxicity studies (Bartczak et al., 2015; Farkas et al., 2011; Filipe et al., 2010; Gallego-Urrea et al., 2011, 2010; Hoecke et al., 2009; Luo et al., 2013). However, no quantitative study of the ENPs in natural sediment in terms of PSD and PNC has been demonstrated by now.

In this article, for the first time we describe the quantitative application of NTA for exploring the fate of different ENPs in sediment-water systems. The study particles comprised a gold (Au) and iron oxides (Fe₃O₄, magnetite) core with different surface functionalities.

1. Materials and methods

1.1. Synthesis and purification of nanoparticles

Citrate coated gold (AuCIT) ENPs (30 nm) were synthesised following the method reported by Frens (Frens, 1973) with minor modifications, by reducing HAuCl₄ using citric acid at reflux (see more details in Supplementary information). The 11-mercaptoundecanoic acid coated gold ENPs (AuMUDA) were obtained after ligand exchange reaction of citric acid groups from gold particle surface in the AuCIT ENPs suspension (Liu et al., 2012a). Different magnetite ENPs (8 nm) were synthesised by a co-precipitation method in alkaline water in the presence of diethyl aminoethyl dextran, dextran (Xu et al., 2005) and sodium citrate reflux at pH 2 respectively. The surface of the ENPs was modified using different coatings and functional groups (Mejias et al., 2008), including diethyl aminoethyl dextran (MDEXN), dextran (MDEX) and citric acid (MCIT). The particle sizes of all samples were measured using TEM (JEOL, FX 2100). The hydrodynamic particle sizes were measured by NTA following dispersion in water. The Zeta potential (ξ -potential) of the magnetic ENPs was recorded using a DLS system (Malvern, Zetasizer Nano ZS). The mass concentration was determined by ICP-MS or AAS.

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