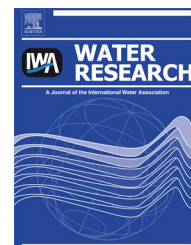


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Long-term colloidal stability and metal leaching of single wall carbon nanotubes: Effect of temperature and extracellular polymeric substances

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ABSTRACT

Long term (90 day) stability, aggregation kinetics in the presence and absence of natural organic materials (NOM), and metal leaching of five commercial single wall carbon nanotubes (SWCNTs) in waters (e.g. freshwater, seawater, stormwater, wastewater, and groundwater) were studied, as well as the effect of temperature on SWCNT stability and metal leaching. Zeta (ζ) potential of SWCNT decreased in magnitude with increase in temperature. In wastewater, SWCNT sedimented from the water column to below detectable levels after 30 days when kept at 40 °C, but at 20 °C 19% suspension was still observed after the same exposure time. Addition of 0.1 mg-C L⁻¹ EPS shifted the critical coagulation concentration (CCC) of SRNOM-stabilized SWCNT from 15 mM to 54 mM NaCl via additional electrostatic and possibly steric stabilization. Attachment efficiencies (α) of SWCNT in waters ranged from ~0.001 in DI with 10 mg L⁻¹ SRNOM to 1 in seawater. However, sedimentation of SWCNT in seawater (and other high ionic strength conditions) was not as fast as expected due to improved buoyancy and/or drag. Purified forms of SWCNTs exhibited better dispersibility and stability in most waters, but as expected, the total metal leached out was higher in the raw variants. Metal leaching from CNT in these studies was controlled by metal and water chemistries, CNT pretreatment, leachable metal fraction, exposure time, and presence of NOM.

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1. Introduction

Carbon nanotubes (CNTs) are insoluble and not easily biodegradable (Lam et al., 2004), hence, they tend to persist in the environment. In order to assess the long-term impact of CNTs

released into surface waters, there is a need to understand how CNTs will partition between surface waters and sediment phases (Schwyzer et al., 2012). The hydrophobic surfaces of CNTs makes them interact readily with natural organic matter (NOM), which is abundant in the environment (Saleh et al., 2010). Hyung et al. (2007) demonstrated improved CNT

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stability in the presence of Suwanee River natural organic matter (SRNOM). In water bodies with significant amounts of NOM, such as humic acid, alginate or extracellular polymeric substances (EPS), the stability of CNTs may be enhanced. As such, CNTs may be mobile in natural aquatic systems, and available to pelagic organisms (Schwyzer et al., 2012). EPS are composed of a wide range of organic polymers such as polysaccharides, proteins, nucleic acids and phospholipids excreted by microorganisms (Flemming et al., 2007); and they may affect the fate and transport of engineered nanomaterials (ENMs) in natural systems (Miao et al., 2009).

Colloidal stability of CNTs in aqueous media has been investigated (Lin et al., 2010; Schwyzer et al., 2011, 2012). Schwyzer et al. (2012) investigated the stability of CNTs in aqueous media and concluded that surface functionalization and size correlates with stabilization. Bennett et al. (2013) monitored the dispersibility and stability of SWCNTs in freshwaters for 72 h and found that stability varies widely based on physicochemical properties of SWCNTs. However, long-term stability of CNTs in natural waters has not been examined. More so, the potential effect of temperature and EPS on stability of CNTs has not been reported to date.

As-prepared CNTs usually contain a significant amount of impurities, including metal catalysts (Bennett et al., 2013; Ge et al., 2012). These impurities are commonly removed in order to obtain purified CNTs. However, some of the metals are protected by graphitic shells, and may remain even after rigorous purification processes (Ge et al., 2011). Residual metal impurities may be mobilized upon introduction of CNTs into aquatic systems (Bennett et al., 2013; Ge et al., 2012). The water chemistry may or may not be favorable for dissolution of metal catalysts attached to CNTs (Ge et al., 2012) so leaching of metals may vary widely in different waters. Metal leaching from CNTs when dispersed in natural waters has only been investigated on a short term basis and no significant toxicity to algae was reported within the study period (Bennett et al., 2013). However, a 14-day study (Mwangi et al., 2012) reported that nickel leached out from CNTs suspensions at levels that were toxic to amphipods. As such, it is important to quantify the levels of metal impurities that may leach out of CNTs in natural waters over a long period of time.

The goals of this study were to experimentally measure the fate of SWCNT, applied from dry powders to natural waters, and investigate:

1. Long-term stability and partitioning of SWCNTs;
2. Long-term leaching of metals from SWCNTs; and
3. Effect of temperature variations on SWCNT surface characteristics, stability, and metal leaching.

2. Materials and methods

2.1. Materials

Five SWCNTs were used for dispersibility and stability studies, which include (1) raw (HP-R) and (2) purified (HP-P) forms of high-pressure carbon monoxide SWCNTs (HiPCo, Nano-Integris, IL); (3) raw (P2-R), and (4) purified (P2-P) forms of arc

discharge SWCNTs (Carbon Solutions, CA); and (5) SG65, a purified SWCNT prepared using the CoMoCat process (SouthWest NanoTechnologies, OK). All five SWCNTs were used as received. P2-P was selected as a model commercial SWCNT, and was used for all the temperature-dependent and aggregation studies. Some physicochemical properties of these SWCNTs have been described previously (Bennett et al., 2013). Additional characterizations done in this study include zeta (ζ) potential analysis (Zetasizer Nano-ZS90, Malvern, UK), transmission electron microscopy (FEI Titan 300 kV FEG TEM), and scanning electron microscopy (FEI XL30 Sirion equipped with an EDAX APOLLO X probe for energy-dispersive X-ray spectroscopy, EDS).

Waters used for this study include NANOpure water with or without 10 mg L⁻¹ SRNOM (denoted DI and DINOM respectively), freshwater with 0.1 mg L⁻¹ (FW0.1) or 1.0 mg L⁻¹ SRNOM (FW1.0), groundwater (GW), lagoon (LW), stormwater (STM), wastewater (WW), and seawater (SEA). Basic characteristics of the waters are shown in Table 1 and additional information is provided in the Supplementary material. Soluble EPS was isolated from an axenic culture of a phytoplankton, *Isochrysis galbana*. Details on EPS isolation are in the Supplementary material. EPS was characterized by measuring carbohydrate and protein concentrations using anthrone method (Morris, 1948), and modified Lowry Protein Assay Kit (Pierce Biotechnology) respectively. Hydrodynamic diameter (HDD) of EPS was determined using the Zetasizer Nano-ZS90.

2.2. Effect of temperature and natural organic materials on zeta (ζ) potential

10 mg L⁻¹ P2-P suspensions were made in the waters by probe-sonication with a Misonix Sonicator S-4000 (QSonica LLC, Newtown, CT). Sonication was done in ice-bath to reduce defect. ζ potential of an aliquot was then determined from 1 to 60 °C in DTS 1060 cells (Malvern) using the temperature controlled chamber of the Zetasizer Nano-ZS90. At each temperature, a 120 s equilibration period was allowed before ζ potential data were collected in triplicate measurements—with each measurement reflecting 12–15 runs. Trend experiments were not done in LW, SEA and WW because the ionic strength (IS) of those conditions was too high for the cells to function properly across the temperature range.

To see the effect of NOM on ζ potential, SWCNT stock suspensions were made with SRNOM or EPS or neither (see Section 2.3 for details). Aliquots were taken from the three stock suspensions, diluted to make a final [SWCNT] of 10 mg L⁻¹, and analyzed for ζ potential at different NaCl concentrations. All ζ potential measurements were done at pH 7.

2.3. Aggregation and sedimentation kinetics

Stabilized SWCNT stock suspensions (100 mg L⁻¹) were made by dispersing P2-P in NANOpure water (Barnstead) in ice-bath by probe sonication using the Misonix Sonicator S-4000 in the presence of 3 mg L⁻¹ SRNOM. Sonicated SWCNT stock was allowed to stand overnight and stable aqueous phase was removed for characterization and analyses. SWCNT stock was diluted by a factor of 10 for aggregation and sedimentation

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