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Effects of ionic strength and temperature on the aggregation and deposition of multi-walled carbon nanotubes

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ABSTRACT

The aggregation and deposition of carbon nanotubes (CNTs) determines their transport and fate in natural waters. Therefore, the aggregation kinetics of humic-acid treated multi-walled carbon nanotubes (HA-MWCNTs) was investigated by time-resolved dynamic light scattering in NaCl and CaCl₂ electrolyte solutions. Increased ionic strength induced HA-MWCNT aggregation due to the less negative zeta potential and the reduced electrostatic repulsion. The critical coagulation concentration (CCC) values of HA-MWCNTs were 80 mmol/L in NaCl and 1.3 mmol/L in CaCl₂ electrolyte, showing that Ca²⁺ causes more serious aggregation than Na⁺. The aggregation behavior of HA-MWCNTs was consistent with Derjaguin–Landau–Verwey–Overbeek theory. The deposition kinetics of HA-MWCNTs was measured by the optical absorbance at 800 nm. The critical deposition concentrations for HA-MWCNT in NaCl and CaCl₂ solutions were close to the CCC values, therefore the rate of deposition cannot be increased by changing the ionic strength in the diffusion-limited aggregation regime. The deposition process was correlated to the aggregation since larger aggregates increased gravitational deposition and decreased random Brownian diffusion. HA-MWCNTs hydrodynamic diameters were evaluated at 5, 15 and 25°C. Higher temperature caused faster aggregation due to the reduced electrostatic repulsion and increased random Brownian motion and collision frequency. HA-MWCNTs aggregate faster at higher temperature in either NaCl or CaCl₂ electrolyte due to the decreased electrostatic repulsion and increased random Brownian motion. Our results suggest that CNT aggregation and deposition are two correlated processes governed by the electrolyte, and CNT transport is favored at low ionic strength and low temperature.

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Introduction

Carbon nanotubes (CNTs) have attracted significant attention due to their remarkable physiochemical properties. They have potential electronic, energy, medical, and environmental applications (Barinov et al., 2009; De Volder et al., 2013; Mu et al., 2009; Ali et al., 2015). However, wide application will cause

the release of CNTs into the ecosystem, which may lead to environmental risk. CNTs have been reported to be toxic to microbes, plants, fish, and mammals (Pasquini et al., 2012; Ma et al., 2015; Cheng and Cheng, 2012; Zhang et al., 2012a). Therefore, the transport of CNTs in the ecosystem has become a hot research field, which is related to both CNT application and evaluation of its environmental hazard.

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Aggregation and deposition processes determine the stability of CNTs in suspension, which is important to estimate their transport and fate in the aqueous environment. Natural organic matter (NOM) is a ubiquitous component of natural waters and plays a key role in the aggregation and deposition of particles and colloids in environmental systems. Adsorbed NOM prevents CNT aggregation through electrostatic repulsion and steric effects (Hyung et al., 2007; Chowdhury et al., 2012), therefore increases the colloidal stability. Cations and ionic strength also have significant influence on particle aggregation. Divalent cations can form cation bridges between negatively-charged particles, which accelerate particle aggregation (Chen and Elimelech, 2007). Increases in ionic strength will compress the electric double layer of particles, and therefore induce particle aggregation (Amal et al., 1992; Chen and Elimelech, 2007). The larger aggregates usually are faster to deposit due to increased gravity and decrease random Brownian diffusion (Petosa et al., 2010). However, little experimental work to date has investigated the connection between particle aggregation and deposition.

The temperature changes in the natural aqueous system may also affect CNT aggregation and deposition. High temperature corresponds to energy input into a particle suspension, which may lead to disruption of interaction forces, increased random Brownian motion of nanoparticles and chances of particle-particle collision, and decreased zeta potential (Zhang et al., 2012b). Although temperature has been considered to be important for nanoparticle stability and transport (Grasso et al., 2002; Karimian and Babaluo, 2007; Li and Elimelech, 2004), the existing studies have not obtained consistent results due to the multiple factors related to temperature. Elevated temperature has been reported to decrease the aggregation kinetics of CeO₂ nanoparticles (NPs) (Zhang et al., 2012b). However, in a study on single wall carbon nanotubes, the colloidal stability decreased significantly from 20 to 40°C (Adeleye and Keller, 2014). Therefore, the relationship between CNT colloidal stability and temperature needs to be further addressed, especially in the presence of NOM. Understanding the effects of temperature is helpful to estimate CNT transport in different seasons.

In this study, the most commonly produced and applied pristine multi-walled carbon nanotubes (MWCNTs) are selected due to their high global production and their inevitable release into the environment. Production of MWCNTs will reach nearly 12,766 metric tons in 2016, mainly driven by use in polymers and composites for automotive components, structural aerospace parts, lithiumion batteries, and some other markets (Jeong et al., 2015). Humic acid (HA) is a major organic constituent of many streams, lakes, and ocean water (Stevenson, 1994). Therefore, the MWCNTs are treated with HA to simulate their interaction with NOM in natural waters. To explore the relationship between aggregation and deposition, and to investigate the effects of ionic strength and temperature, the MWCNT aggregation and deposition experiments are performed in varied electrolytes at different temperatures. MWCNT aggregation was measured by dynamic light scattering (DLS) and deposition was measured by optical absorbance at 800 nm. Our findings will be important to understand CNT transport in the natural waters and to estimate its environmental risk.

1. Experimental section

1.1. Materials

Pristine MWCNTs with length of 1–10 μm were purchased from Chengdu Organic Chemistry Co. Ltd., Chinese Academy of Sciences. The specific surface area (SSA) of MWCNTs was 127 m²/g measured by the multi-point Brunauer–Emmett–Teller method.

HA extracted from leonardite was purchased from Aladdin Reagent Co. The commercial HA was characterized in our previous study (Hu et al., 2014). In summary, it contains 40.2% C, 3.76% H, 49.1% O, 0.99% N, 0.86% S, and a high content of aromatic groups and carboxylic groups. Its aromaticity is 47.92% reflected by solid state ¹³C nuclear magnetic resonance spectroscopy (Hu et al., 2014). HA powder was dissolved in deionized (DI) water to 1000 mg/L and was sonicated by a KQ3200E sonicator (Kunshan Ultrasonic Instruments, China) for 30 min. The HA solution was filtered via a 0.22 μm syringe filter to remove undissolved residues. The mass loss after filtration is less than 1.5%. The quantitative standard curve of HA solution was obtained by a ultraviolet-visible (UV-vis) Spectrometer (TU-1900, Beijing Purkinje General Instrument) at 210 nm.

DI water (18.25 MΩ/cm) was used in experiment. The NaCl (1 mol/L) and CaCl₂ (0.1 mol/L) electrolyte stock solutions were prepared and filtered through a 0.22 μm syringe filter before use.

1.2. Preparation of HA-treated MWCNT suspension

The HA-treated MWCNTs (HA-MWCNTs) were prepared by a modified method based on previous studies (Li and Huang, 2010; Saleh et al., 2010). To prepare HA-MWCNTs, 6 mg MWCNTs was mixed with HA solution (100 mg/L, 30 mL). The mixture was adjusted with 0.1 mol/L HCl/NaOH to pH 5–6, and was shaken (120 r/min, 25°C) for 48 hr to allow HA to adsorb on MWCNTs. The HA-MWCNTs were centrifuged (3160 ×g, 30 min) to remove unadsorbed HA in supernatant, then washed twice with DI water. The amount of adsorbed HA on MWCNTs was 70.5 ± 7.5 mg/g, which was calculated from the HA equilibrium concentration in supernatant and the HA loss in the washing process. After washing, HA-MWCNTs were dispersed in DI water by mild sonication (40 kHz, 25°C) for 2 hr. Then the HA-MWCNT dispersion was centrifuged (3160 ×g, 30 min) to obtain a stable suspension (20 mg/L). The suspension was adjusted to pH 7.2 with 0.1 mol/L HCl/NaOH and kept as a stock suspension. After removing MWCNTs with a 0.22 μm syringe filter, the free HA in stock suspension was measured to be less than 2 mg/L by a UV-vis spectrophotometry. Such HA treatment simulated the condition that MWCNTs were fully interacted with sufficient amount of HA.

1.3. Quantification of HA-MWCNT suspension

The HA-MWCNT suspension was quantified according to previous methods (Yu et al., 2007; Yang et al., 2013). MWCNTs (1 mg) were mixed with 40 mL of 4 mg/mL sodium dodecyl sulfate (SDS) solution and were sonicated at 25°C for 2 hr to obtain a uniform stock suspension. The stock suspension was

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