



Removal of sodium dodecyl sulfate surfactant from aqueous dispersions of single-wall carbon nanotubes



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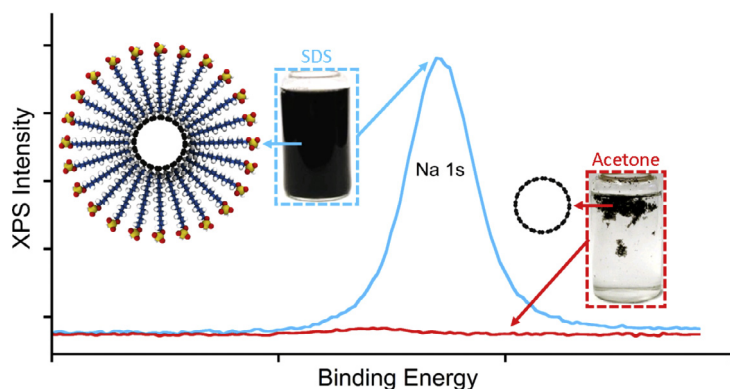
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GRAPHICAL ABSTRACT



ARTICLE INFO

Article history:

Received 20 December 2016

Revised 27 January 2017

Accepted 31 January 2017

Available online 2 February 2017

Keywords:

Dispersion
Solvent interaction
SWCNT purity
Surfactant removal

ABSTRACT

A reagent-based treatment method was developed for the removal of sodium dodecyl sulfate (SDS) from aqueous dispersions of single-wall carbon nanotubes (SWCNTs). Based on a survey of various reagents, organic solvents emerged as the most effective at interrupting the SDS:SWCNT interaction without producing deleterious side reactions or causing precipitation of the surfactant. Specifically, treatment with acetone or acetonitrile allows for the facile isolation of SWCNTs with near complete removal of SDS through vacuum filtration, resulting in a 100x reduction in processing time. These findings were validated via quantitative analysis using thermogravimetric analysis, Raman spectroscopy, 4-point probe electrical measurement, and X-ray photoelectron spectroscopy. Subsequent thermal oxidation further enhances the purity of the reagent treated samples and yields bulk SWCNT samples with >95% carbonaceous purity. The proposed reagent treatment method thus demonstrates potential for large volume SWCNT processing.

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1. Introduction

Amphiphilic surfactants have facilitated the use of single-wall carbon nanotubes (SWCNTs) in a number of applications, enabling

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the exploration of their unique physical and electrical properties. The limited solubility of SWCNTs in water makes them difficult to work with, necessitating the use of surfactants and ultrasonication to form stable aqueous dispersions. Sodium dodecyl sulfate (SDS) is the most commonly employed surfactant in applications such as spectroscopy [1–5], chirality separations [6–8], and device fabrication (*i.e.*, transparent conducting films [9,10], organic photovoltaics [11], batteries [12], etc.). However, the presence of residual SDS on the surface of the SWCNTs makes it difficult to probe their intrinsic properties and can require additional post-processing purification for some applications.

Early reports indicated that SDS could be removed from SWCNTs via vacuum filtration and copious water rinsing [13,14]. The amount of water rinsing was typically determined using visual indicators when bubbling of the surfactant through the filter membrane stopped; however, there has been insufficient evidence of the resulting SWCNT purity through quantitative analysis for this method. In fact, recent literature demonstrates that simple water rinsing alone leaves behind up to 15 wt% residual SDS in SWCNT thin-films prepared by this technique [12]. As such, the addition of thermal oxidation and/or annealing after sample processing facilitates the removal of residual SDS from water rinsed SWCNT thin-films and bulk papers [15–18], yet complete SDS removal with thermal treatments alone has not been demonstrated. Alternate approaches have examined the removal of residual SDS and other common surfactants by soaking SWCNT samples in concentrated nitric acid, which is proposed to remove the residual SDS, as well as defective SWCNTs from the sample [9–12,19]. Furthermore, acidification of the surfactant salt with HCl creates a byproduct that can be solubilized and washed away with ethanol [20]. More sophisticated techniques have focused on the decomposition of organic compounds, such as surfactants, through the use of the Fenton reaction [21]; however, as with the acid treatments this is a time consuming process that has not been shown to fully remove the residual SDS from the SWCNTs.

In the current work, a facile and scalable reagent-based method of surfactant removal is developed to isolate SWCNTs from aqueous SDS-based dispersions. Various reagents are evaluated for their ability to interrupt the SDS:SWCNT interaction in solution, with the goal of precipitating the SWCNTs for easy isolation via vacuum filtration. The resulting bulk SWCNT papers were characterized using scanning electron microscopy (SEM), Raman spectroscopy, thermogravimetric analysis (TGA), 4-point probe electrical measurements, and X-ray photoelectron spectroscopy (XPS) to evaluate the purity of the reagent treated samples compared to a control that was filtered from SDS and rinsed only with water. A portion of each sample is subsequently exposed to thermal oxidation treatment in air, and the characterization is repeated to evaluate any enhanced purity of the reagent treated SWCNT samples. A distinct relationship is observed between SWCNT purity and reagent composition, and an optimized purification protocol is developed for the removal of SDS from SWCNTs.

2. Experimental

2.1. SWCNT synthesis and preparation of P-SWCNT control sample

Single-wall carbon nanotubes (SWCNTs) were synthesized in-house via pulsed laser vaporization employing an Alexandrite laser (755 nm) in argon carrier gas at 1150 °C. The laser pulse was rastered over the surface of a graphite (Alfa Aesar, Graphite Flake, median 7–10 μm, 99% metal basis) target doped with 3% w/w Ni (Sigma-Aldrich, <1 μm, 99.8% trace metals basis) and 3% w/w Co (Sigma-Aldrich, 2 μm, 99.8% trace metals basis) [22]. The as-produced SWCNT soot was purified of metal catalyst and

carbonaceous impurities using previously established acid reflux and thermal oxidation (ramp-stop at 10 °C/min to 520 °C) procedures [23,24]. The SWCNT sample was further purified by soaking in 37% HCl for 10 min, rinsed with copious amounts of deionized (DI) H₂O, then soaked for an additional 1 h in 37% HCl to remove residual Ni and Co catalyst. The sample was rinsed with DI H₂O, then thermally oxidized as described previously to remove residual acid and carbonaceous impurities. The purified SWCNT material (P-SWCNT) serves as the experimental control for all subsequent reagent-treated SWCNT samples.

2.2. SWCNT characterization

The purity of the SWCNT bulk papers was analyzed qualitatively via scanning electron microscopy (Hitachi S900 Field Emission SEM, 2 kV accelerating voltage) and quantitatively using optical absorption spectroscopy (Perkin-Elmer Lambda 900), Raman spectroscopy (Jobin Yvon Horiba LabRam Spectrometer, 1.96 eV laser energy), thermogravimetric analysis (TA Instruments TGA Q5000, Balance Purge: N₂(g) 20 mL/min, Sample Purge: Air 20 mL/min, Ramp Rate: 10 °C/min), and X-ray photoelectron spectroscopy (ThermoScientific Monochromatic Al k-alpha source (1487 eV), 50 eV pass energy, 400 μm spot). Electrical characterization was performed via the 4-point probe van der Pauw method using a Signatone H100 Series probe station affixed with a NI PXI-4130 Power SMU to supply the current and PXI-4171 for voltage measurement.

2.3. Preparation of SDS-SWCNT and H₂O-SWCNT control samples

P-SWCNTs were dispersed in 840 mL of an aqueous solution of 2.0 wt% sodium dodecyl sulfate (SDS, Sigma-Aldrich, ACS reagent, ≥99.0%) with a SWCNT concentration of 50 μg/mL via bath (3 h) and horn (5 h) ultrasonication in an ice-water bath. A 120 mL aliquot was removed, and the SWCNTs were isolated via vacuum filtration onto a PTFE membrane (0.1 μm pore size). As a control, the SWCNTs were rinsed with 50 mL of DI H₂O and remained under vacuum in the filtration apparatus to dry for 1 h, yielding a bulk paper (SDS-SWCNT) with an areal mass density of 0.6 mg/cm². For consistency, the SWCNT mass and areal density was fixed for all additional SWCNT samples fabricated in the current study. To control for the effect of processing on the SWCNT physical and electrical properties, a second control sample was prepared whereby P-SWCNTs were ultrasonicated in DI H₂O (no surfactant) using the same concentration, processing times, and conditions as described above. This sample (referred to as H₂O-SWCNT) was subsequently filtered, rinsed, and dried in a manner consistent with the SDS-SWCNT sample. After releasing the SDS-SWCNT and H₂O-SWCNT samples from the filter membrane, both samples were further dried in a vacuum oven at 100 °C for 1 h.

2.4. Preparation of HCl-SWCNT control sample

A third control sample (referred to as HCl-SWCNT) was prepared using a previously reported procedure for removing sodium deoxycholate from SWCNTs [20]. In short, a 120 mL aliquot of SWCNTs in 2.0 wt% SDS was removed from the stock and mixed with an equal volume of 6 M HCl. The mixture was bath sonicated for 1 h, then vacuum filtered onto a PTFE membrane and rinsed with 50 mL of DI H₂O. The sample was redispersed in ethanol via bath sonication for 1 h. The resulting dispersion was heated to 80 °C in a water bath for 1 h, and subsequently filtered onto a PTFE membrane to isolate the treated SWCNTs. The HCl-SWCNT sample was rinsed with fresh ethanol and remained under vacuum in the filtration apparatus to dry for 1 h, then dried at 100 °C for 1 h in a vacuum oven after releasing from the filter membrane.

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