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Sodium dodecyl benzene sulfonate for single-walled carbon nanotubes separation in gel chromatography

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ABSTRACT

Though sodium dodecyl benzene sulfonate (SDBS) exhibited stronger dispersing ability for single-walled carbon nanotubes (SWCNTs) in comparison to sodium dodecyl sulfate (SDS). SDBS was not reported to be successfully employed in SWCNTs separation. In this work, we report that SDBS dispersed SWCNTs can be loaded onto Sephacryl gel S200 and these retained SWCNTs can be selectively eluted out using SDBS eluent with various concentrations and its mixture composition with SDS.

1. Introduction

Since Kappes et al. firstly reported using Sephacryl gels for single-walled carbon nanotubes (SWCNTs) separation with sodium dodecyl sulfate (SDS) as dispersant and eluent [1], SDS based gel chromatography for SWCNTs separation has been well developed for single-chirality, and enantiomeric SWCNT purification with the assistance of multiple cycling and temperature control [2–4]. More advanced development revealed that the mixture surfactants of SDS and sodium cholate (SC) [5,6] or sodium deoxycholate (DOC) [7] could selectively release the specific SWCNT species. Gel has been excellent separation media for biological macromolecules like DNAs, RNAs, proteins, and even virus and bacteria. In these work, surfactants encapsulated SWCNTs were hypothesized to be captured by methylene bis(acrylamide) cross-linkers [8] or hydroxyl group of Dextran [9]. Recently, we proposed the 15-crown-5 ether like microstructures (5 OH groups in proximity) in Sephacryl gels might loosely chelate Na^+ ions and negatively charged dodecyl sulfate (DS) moieties swung around based on electrostatic principle [10].

Sodium dodecyl benzene sulfonate (SDBS) is a common surfactant for dispersing SWCNTs stronger than SDS [11]. Ju et al. even reported SDBS exhibited stronger binding affinity to SWCNTs sidewalls than SC in their displacement experiments for flavin mononucleotide (FMN) adsorbed on SWCNTs surfaces [12]. This strong binding affinity of SDBS is ascribed to a benzene ring inserted between dodecyl and sulfonate of SDS that preferably forms π-π interactions on SWCNT sidewalls [13]. Such π-π interactions could enable the SDBS dispersed SWCNTs with little diameter selectivity [14]. Which might result in no observed fractionation of SWCNTs in density gradient ultracentrifugation [15]. In gel chromatography for SWCNT separation, as our best knowledge, no work was reported using SDBS as dispersant and eluent.

In this research, the SDBS was used to substitute SDS for dispersing SWCNTs, for loading SDBS dispersed SWCNTs on Sephacryl gels, and for eluting the retained SWCNTs out from Sephacryl gels using various concentrations and mixture compositions with SDS. The eluted solutions were characterized for Visible-Near Infrared (Vis-NIR) absorption spectra using NS3 Applied Nano Spectralyzer. The spectral results demonstrate that SDBS can be used for SWCNTs separation in gel chromatography alone or mixed with SDS. The stronger SWCNTs dispersing ability and relatively lower price of SDBS in comparison to SDS could improve the cost effectiveness for SWCNTs separation.

2. Experimental details

2.1. Materials

Sodium dodecyl sulfate (SDS), sodium cholate (SC), sodium dodecyl benzene sulfonate (SDBS) were purchased from Sigma- Aldrich and used as received. Sephacryl gel S200 was purchased from GE health. Single-walled carbon nanotubes (SWCNTs) raw powder was produced by Rice University Mark III high pressure carbon monoxide reactor using less catalyst in a yield of 1 g per hour. The raw SWCNTs were used for the extraction of (6,5) SWCNT using gel permeation chromatography.

2.2. Measurement

All Visible Near-Infrared absorption spectra of SWCNTs dispersed in

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different surfactant systems were measured on NS3 Applied Nano Spectralyzer at ambient condition.

3. Results and discussion

We have extensively researched on SWCNTs separation using gel chromatography [16–18]. Following the work reported from Kataura and Strano's research groups [2–4, 19–21], we concluded the importance of the saturation of targeted SWCNT species on gels, which directly related to the abundance of targeted SWCNT species in supernatants, based on our SWCNTs separation development. The distribution of SWCNTs in supernatants is determined by the types and batches of SWCNTs, sonication power and times, centrifugation speed and times [19]. The SWCNT diameter-selectivity is largely related to the composition and concentration of surfactant systems [2,3]. Generally, the lower concentration SDS solution is preferred for the larger diameter SWCNT purification. For examples, the 2% (weight%) and 3% SDS solution is good for (6,5) SWCNT separation [2–4,19], and 0.5% SDS solution is good for (7,6) SWCNT enrichment [20]. Even single-chirality and enantiomeric metallic SWCNT was separated using gel chromatography with 0.3% SDS [21]. The specific surfactant compositions such as the 15/1 SDS/DOC for (6,5) SWCNT independent of the concentrations [7], 0.5% SDS/0.5% SC/0.02% DOC for (7,3) SWCNT, 1% SDS/0.5% SC/0.13% DOC for (9,4) SWCNT, and (10,3) SWCNT were reported [5,6]. The surfactant compositions are very mystery, some anomalous phenomena were observed [22]. To remain constant temperature is critical to achieve consistent separation. The low temperature around 16–18 °C is indeed helpful to achieve high pure smaller diameter SWCNTs [3]. The pore sizes of Sephacryl gels are important [1,23,24], but S200 and S300 gels are similar for small diameter SCWNTs separation.

For the purification of (6,5) SWCNT, the 5% SDS, 5% SC, and 2% SDS/0.05% SC and 2% SDS/0.5% SC were used to elute out the retained (6,5) SWCNT in Sephacryl S200 Gels in parallel. The obtained solutions were characterized for Vis-NIR absorption using NS3 Applied Nano Spectralyzer. Fig. 1 shows the Vis-NIR absorption spectra of eluted SWCNTs normalized at the first optic transition S_{11} absorption peak of (6,5) SWCNT [25] using 5% SC (black curves), 5% SDS (red curve), 2% SDS/0.5% SC (green curves), and 2% SDS/0.05% SC (blue curve) respectively. Because the loading solution was 2% SDS dispersed SWCNTs solution. The two portions eluted out using 5% SC were significantly different. The first portion (black curve) with S_{11} absorption peak of (6,5) SWCNT is between 2% SDS/0.5% SC (blue curve) and 2% SDS/0.05% SC (green curves), indicating SDS was the major component [25]. Additionally, its absorption spectrum resembles those of solutions eluted with 5% SDS (red curve) and 2% SDS/0.05% SC (greens curves) eluents. The S_{11} absorption peak of (6,5) SWCNT in second portion is similar to (6,5) SWCNT dispersed in 5% SC, implying that the second portion was majorly with 5% SC species [26]. Moreover, the large diameter SWCNTs with absorbance peaked at 1135 nm was eluted out. The spectra elicit that the dominant large diameter SWCNTs were eluted out using the eluent of 5% SC. The absorption spectra of solution eluted with 2% SDS/0.05% eluents are similar to that eluted with 5% SDS eluent and the first portion eluted with 5% SC. In these absorption spectra, the absorbances are dominated by (6,5) SWCNT specie with large diameter SWCNTs species much weaker than the second portion solution eluted out using 5% SC. Constrastly, the Vis-NIR absorption spectrum of solution eluted out with 2% SDS/0.5% SC represents the best purity of (6,5) SWCNT separation, indicating a specific SDS/SC mixture surfactant system selective to (6,5) SWCNT.

Similarly, the 2% SDS loaded SWCNTs were eluted using 2% SDS/0.5% SDBS, 2% SDS/0.25% SDBS, and 2% SDS/0.05% SDBS respectively in parallel. The obtained solutions were measured on NS3 Applied Nano Spectralyzer. The obtained Vis-NIR absorption spectra were normalized at the S_{11} peak wavelength of (6,5) SWCNT and were presented in Fig. 2. Same as SDS/SC system, the solution eluted with 2% SDS/0.5% SDBS (blue curve) exhibited the clear spectrum implying more (6,5) SWCNT enriched with less contamination of large diameter SWCNT species as observed in the absorption spectra (peaked at 1021 nm and 1138 nm) of solutions eluted out with 2% SDS/0.25% SDBS (dark red) and 2% SDS/0.05% SDBS (dark green). It seems that 2% SDS/0.5% SDBS mixture system can selectively elute out (6,5) SWCNT specie in comparison to other SWCNT species, same as 2% SDS/0.5% SC system discussed in last section.

The SWCNTs dispersed in SDBS solution can be loaded on Sephacryl gel S200 when the concentration of SDBS is less than 0.1%. This is sharply in contrast to SC surfactant for which 0.1% concentration can wash out any retained SWCNTs in Sephacryl gels. Though SDBS was reported to have stronger binding affinity to SWCNTs surfaces than SC [12]. The solution eluted with 0.1% SDBS eluent was characterized for Vis-NIR absorption spectrum on NS3 Nano Applied Spectralyzer. As

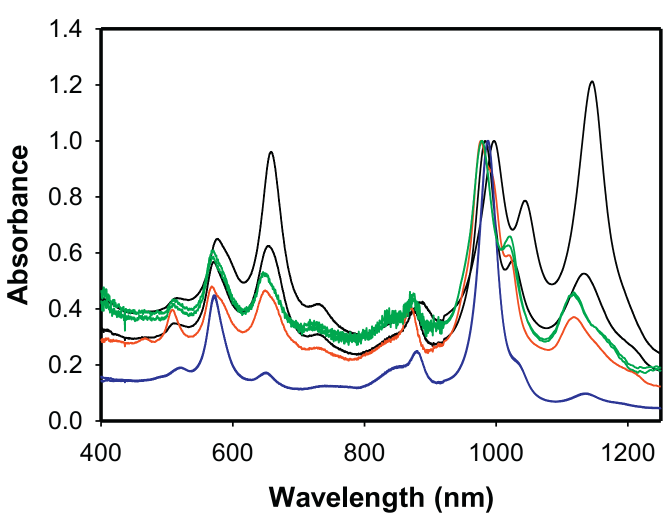

Fig. 1. Vis-NIR absorption spectra of eluted SWCNT species using 5% SC (black curve), 5% SDS (red curve), and 2% SDS/0.05% SC (green curves) and 2% SDS/0.5% SC (blue curve) respectively. These absorption spectra were normalized at first optic transition S_{11} of (6,5) SWCNT. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

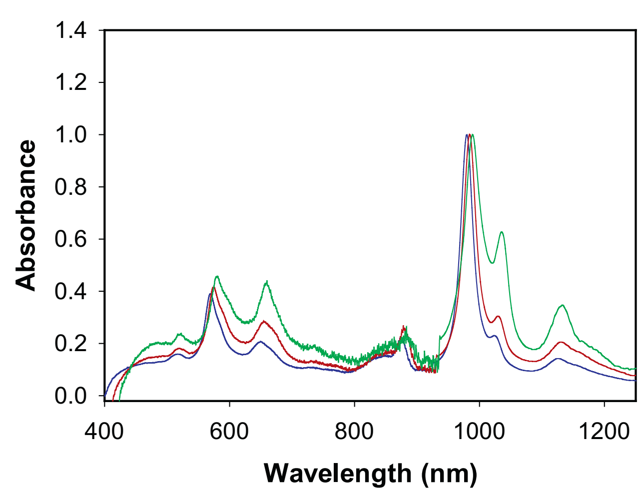

Fig. 2. Vis-NIR absorption spectra of eluted SWCNT species using 2% SDS/0.05% SDBS (blue curve), 2% SDS/0.25% SDBS (dark red) and 2% SDS/0.5% SDBS (dark green) respectively. These absorption spectra were normalized at first optic transition S_{11} of (6,5) SWCNT. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)