

Visible near Infrared Spectroscopic Evidence of the Purification and Dispersion of Single-Walled Carbon Nanotubes

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Abstract

Original Research Article

The application of dispersible single-walled carbon nanotubes in water is required in many fields, such as mechanics, thermology, electricity, etc. In this paper, single-walled carbon nanotube samples were dispersed in sodium deoxycholate aqueous solution with the assistance of ultrasonic crushing. The effects of centrifugal time, concentration and centrifugal velocity on dispersion were investigated. After centrifugal separation, the dispersible single-walled carbon nanotubes solution in water was obtained. The dispersion behavior of single-walled carbon nanotubes in sodium deoxycholate aqueous solution was further investigated by Uv-visible near infrared spectrum and electrochemical cyclic voltammetry curve. At the same time, characterization results of Uv-visible near infrared spectrum show that the purity of single-walled carbon nanotubes was improved. The cyclic voltammetry curves of single-walled carbon nanotubes after purification and dispersion were tested by electrochemical method, and the electron transfer behavior of single-walled carbon nanotubes after purification and dispersion was investigated.

Keywords: Single-walled carbon nanotubes; Uv-visible near infrared spectrum; Dispersion; Sodium deoxycholate.

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INTRODUCTION

Single-wall carbon nanotubes (SWCNTs) have unique electrical properties, mechanical properties and optical properties [1]. But due to the van der Waals forces between the SWCNTs bundles, SWCNTs are commonly bunched, and their insolubility and difficult dispersion in aqueous media, which further severely limited their wide application in many fields. Therefore, the preparation of highly dispersible SWCNTs in water is a meaningful work.

In recent years, many researches have used various organic and inorganic solvents, surfactant molecules and biological molecules as dispersants to solve this problem. Materials, such as surfactants, ionic liquids, biomolecules, and polymers commonly were used to disperse SWCNTs. Syrgiannis, Z. found that polyoxometalates(POM) had different dispersing capacities for small diameter SWCNTs, this result could be derived from the interaction POM and SWCNTs [2]. Ranran Wang reported that SWCNTs were dispersed by pristine DNA, which had better dispersion performance for SWCNTs, and the DNA/SWCNTs were prepared transparent conductive films on PET substrates, the films with high performance and good stability had been obtained [3].

Matsumoto, K. used ketone solvents to disperse SWCNTs, a stable dispersion system was obtained [4]. A kind of polymer ionic liquids PIL, which synthesized by three steps, was used to disperse SWCNTs, SWCNTs can be dispersed by PIL in DMF, and a sensing materials was made with ionic liquid package SWCNTs, a super sensitive CO₂ sensor is developed, its detection limit is 500 PPT, and the sensor of CO₂ has good selectivity [5]. The dispersion of SWCNTs by biological macromolecules was reported, and the structure and stability of the chiral index (6,5) ssDNA-SWCNTs complex was studied by using the all-atom molecular dynamics simulation method. The dispersion efficiency of SWCNTs by ssDNA, flavonoids mononucleotide surfactants, and polyaminoamine (PAMAM) dendrimers was also compared [6].

In this paper, sodium deoxycholate was used as dispersing agent, single-walled carbon nanotube samples were dispersed in sodium deoxycholate aqueous solution with the assistance of ultrasonic, the dispersion behavior of single-walled carbon nanotubes in sodium deoxycholate aqueous solution was further investigated by Uv-visible near infrared spectrum, the electron transfer behavior of single-walled carbon nanotubes after purification and dispersion was

investigated by electrochemical cyclic voltammetry curve.

MATERIALS AND METHODS

Materials and reagents

Ac-discharged SWCNTs which were made and purified in laboratory, Ethanol, sodium deoxycholate and potassium hexyanide ferrate were analytical pure.

Instruments and equipment

Supercentrifuge (CP100WX, HITACHI of Japan), vacuum drying oven(DGF-6030, the company of Shanghai yiheng scientific instrument), electronic analytical balance (ALC-310.2, LTD. co. Beijing taike instrument), circulating water multi-purpose vacuum pump(SHB-III A, LTD. co. zhengzhou Great Wall science and trade), constant temperature magnetic stirring water bath(HJ-A2, LTD. co. changzhou maikonuo instrument), Uv-visible spectrophotometer(Uv-3600, HITACHI, Japan), electrochemical workstation(SP-300, Japan electronics).

METHODS

The purification and Dispersion of SWCNTs

SWCNTs were dispersed by sodium deoxycholate in accordance with the method described in literature [7]. Sodium deoxycholate (1 mg) was added in a beaker (150 mL), deionized water (100 mL) was put to dissolve sodium deoxycholate, the solution was dispersed with ultrasonic until the solution was transparent. Then SWCNTs samples (5 mg) were added in the solution of sodium deoxycholate, it was dispersed for 12 h at 5 °C, the solution was put in some centrifuge tubes and put in a high-speed centrifuge, at the conditions of rotating speed was at 20000 rpm, temperature was at 10 °C, the centrifugal separation was for 1 h. At last, the top 80% solution was carefully removed with a syringe, the dispersed solution of

SWCNTs was obtained, the purification of SWCNTs was realized in the meanwhile.

Construction of three-electrode working system

According to the method in literature [8], PBS buffer solution (100 mL, pH=7.4) was prepared, a certain amount of potassium hexanocyanate was added to the buffer solution, a buffer solution (Fe(III)/Fe(II)) was obtained. The glassy carbon electrode was polished by Al_2O_3 , the glassy carbon electrode was washed with alcohol and deionized water in turn. Then the glassy carbon electrode was blow-dried with high purity nitrogen. The purified and dispersed SWCNTs was carefully aspirated onto the glassy carbon working electrode with a micro syringe. The modified working electrode was obtained and placed to dry. Then the auxiliary electrode (platinum wire electrode), reference electrode (saturated calomel electrode) and working electrode modified with purified and dispersed SWCNTs were put into the prepared PBS buffer solution, three-electrode working system was obtained. Cyclic voltammetry characteristics of working electrode modified with sodium deoxycholate-SWCNTs were investigated by electrochemical workstation testing, and electron transport of purified and dispersed SWCNTs was investigated.

RESULTS AND ANALYSIS

The purification and dispersion of SWNTs

The effect of sodium deoxycholate concentration on the dispersion of SWNTs

Ultraviolet near infrared spectroscopy is an important method to investigate the purification and dispersion of SWCNTs, so the purification and dispersion effect of SWCNTs were investigated by the absorption peak intensity of Ultraviolet near infrared spectroscopy. Figure-1 is the Vis-NIR spectra of dispersion solution of SWNTs with different concentration sodium deoxycholate(a) 0.5%, (b) 1.0% and (c) 1.5%.

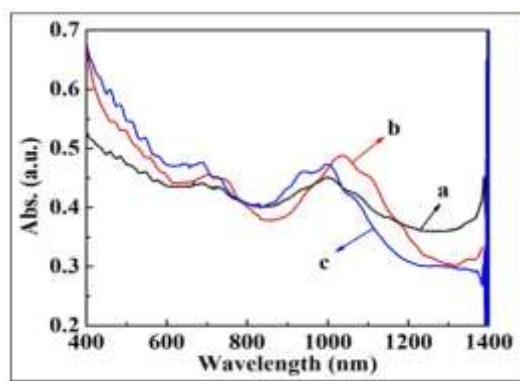


Fig-1: Vis-NIR spectra of dispersion solution of SWNTs with different concentration sodium deoxycholate(a) 0.5%, (b) 1.0% and (c) 1.5%

As is shown in figure 1, the Vis-NIR spectra of the solution of sodium deoxycholate-SWNTs varies with the concentration of sodium deoxycholate. The absorption spectra line (b) presents better resolution and higher effective absorption intensity than those of the

absorption spectra line (a) and (c), the relative absorption peak of line (b) is larger than those of line (a) and (c). So the solution of sodium deoxycholate (1%) has a better dispersion effect on SWNTs. It means the dispersion of sodium deoxycholate towards SWNTs

is related to the concentration of sodium deoxycholate, when the concentration of sodium deoxycholate is 1%, it has the best dispersion ability to SWNTs. When the concentration of sodium deoxycholate is 1%, the purification of SWCNTs was higher, the purification of SWCNTs was realized in the meanwhile.

The effect of centrifugal speed on the dispersion of SWNTs

Figure-2 is the Vis-NIR spectra of dispersion solution of SWNTs with different centrifugal speed (a) 15000 rpm, (b) 20000 rpm and (c) 25000 rpm.

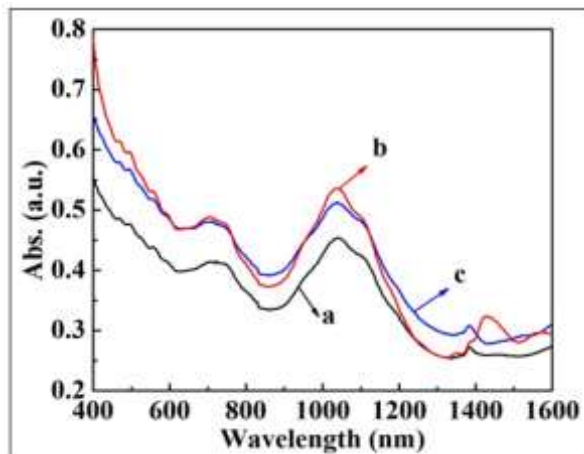


Fig-2: Vis-NIR spectra of dispersion solution of SWNTs with different centrifugal speed (a) 15000 rpm, (b) 20000 rpm and (c) 25000 rpm

As is shown in Figure-2, the absorbance of the solution of sodium deoxycholate-SWNTs varies with the centrifugal speed. The absorption spectra line (b) presents better resolution and higher effective absorption intensity than those of the absorption spectra line (a) and (c), the relative absorption peak of line (b) is larger than those of line (a) and (c). So the centrifugal speed (20000 rpm) has a better dispersion effect on SWNTs. It means the dispersion of sodium deoxycholate towards SWNTs is related to the centrifugal speed, when the centrifugal speed is 20000

rpm, it has the best dispersion to SWNTs. When the centrifugal speed is 20000 rpm, the purification of SWCNTs was higher, the purification of SWCNTs was realized in the meanwhile.

The effect of centrifugal time on the dispersion of SWNTs

Figure-3 is the Vis-NIR spectra of dispersion solution of SWNTs with different centrifugal time (a) 1 h, (b) 1.5 h and (c) 2.0 h.

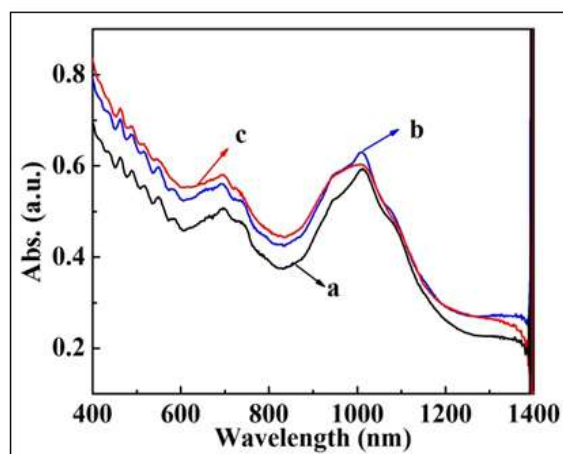


Fig-3: Vis-NIR spectra of dispersion solution of SWNTs with different centrifugal time (a) 1 h, (b) 1.5 h and (c) 2.0 h

As is shown in Figure-3, the absorbance of the solution of sodium deoxycholate-SWNTs varies with the centrifugal time. The absorption spectra line (b) presents better resolution and higher effective absorption intensity than those of the absorption spectra line (a) and (c), the relative absorption peak of line (b) is larger than those of line (a) and (c). So the centrifugal

time (1.5 h) has a better dispersion effect on SWNTs. It means the dispersion of sodium deoxycholate towards SWNTs is related to the centrifugal time, when the centrifugal time is 1.5 h, it has the best dispersion to SWNTs. When the the centrifugal time is 1.5 h, the purification of SWCNTs was higher, the purification of SWCNTs was realized in the meanwhile.

Cyclic voltammetry curves of SWCNTs

Comparing with SWCNTs bundles, dispersible SWCNTs have an excellent electron transport capability, and they are widely used in sensor design [9]. In the experiment, the naked glassy carbon

electrode was modified with SWCNTs before and after dispersion, and the influence of SWCNTs on the electron transfer ability was investigated by analyzing the cyclic voltammetry of the modified glassy carbon electrode in the three-electrode system.

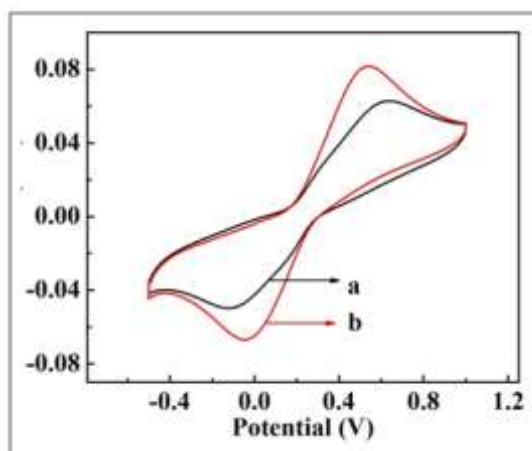


Fig-4: Cyclic voltammetry curve of (a) bare glass electrode and (b) sodium deoxycholate-SWCNTs-bare glass electrode

As is shown in figure 4, curve a is cyclic voltammetry curve of bare glass electrodes in buffer solution of PBS, curve b is cyclic voltammetry curve of glass electrodes modified by SWCNTs in buffer solution of PBS. It can be clearly observed that the peak of curve b is significantly lower than that of curve a, and the peak area of curve b is also significantly greater than that of curve a. This indicated that the electron transport speed of the working electrode was accelerated after the bare glass electrode was modified with porous SWCNTs. Compared with the pore surface area of bare glass electrodes is significantly larger, so the surface of the working electrode modified by SWCNTs presents a better porous structure, which improves the mass transfer effect of the modified working electrode. Thus, it was suggested that the conductivity of the dispersed SWCNTs was improved after sodium deoxycholate dispersion. It was further demonstrated that the dispersibility of SWCNTs in aqueous solution was significantly improved after the action of sodium deoxycholate in aqueous solution. Therefore, the preparation of water-dispersible SWCNTs by this method has potential research value in the field of electrochemistry.

CONCLUSION

SWCNTs were purified and dispersed in sodium deoxycholate solution by ultrasonic fragmentation, and the purified and dispersed SWCNTs in water were obtained by centrifugal separation. When SWCNTs were dispersed by sodium deoxycholate, the sodium deoxycholate formed non-covalent with SWCNTs, which weakened the van der Waals forces between SWCNTs bundles, the dispersibility of SWCNTs was achieved, and the dispersible solution of SWCNTs was obtained. Therefore, it provides a feasible method for obtaining highly purified SWCNTs

that are dispersible in water. CV curve of the dispersion solution of SWCNTs was investigated by three electrode working system, which further demonstrated the good conductivity of the dispersion solution of SWCNTs. Therefore, water-dispersible and high-purity SWCNTs have potential research value in the field of electrochemistry.

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