

# Diameter-dependent voltammetric properties of carbon nanotubes

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## Abstract

Voltammetric properties of electrodes made of multiple-walled carbon nanotubes (MWNTs) with different diameters were investigated by cyclic voltammetry. The results indicate that electrodes made of smaller MWNTs exhibit better voltammetric properties. In addition, electrodes made of mixed MWNTs with large diameter distribution possess the best voltammetric properties. The phenomenon can be explained by the filled-in frame structures formed on the electrode surface.

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## 1. Introduction

Electrodes made using various carbon materials, such as glassy carbon, highly ordered pyrolytic graphite and boron-doped diamond are widely used in electrochemical applications [1–3]. Research has focused on understanding the factors that govern the electron transfer kinetics in carbon electrodes. The effect of solid carbon electrodes' surface structure in directing electron transfer reactions in electrochemistry has been well recognized, and the creation of specific surface structures, through pretreatments, such as plasma activation [4] and functional groups modification [5,6], can accelerate the electron transfer. The surface preparation and the final surface structure are often found to be critical to the performance of electrodes.

Due to their outstanding properties, carbon nanotubes (CNTs) are one of the most attractive nanomaterials in nanotechnology. Many potential applications of CNTs have been investigated, including electron field emitters [7], nanobalance [8], quantum resistors [9], nanoscale mass

conveyors [10], chemical sensors [11], rotational actuators [12], electronic biosensors [13], etc. Based on the specific surface structure of CNTs, applications in electroanalysis have also been studied, and most of them used CNTs to modify the surfaces of traditional electrodes, such as casting CNTs on Pt, Au [14] and glassy carbon electrodes [15], or intercalating MWNTs on graphite electrodes [16]. Some of these CNTs-modified electrodes failed to give well-resolved cyclic voltammograms [14], while others gave well-defined voltammetric responses [15] and can be used to detect low concentration biomolecules [16,17].

In this Letter, we present an alternative strategy by using CNTs film on inert glass substrate as the electrodes. The performance of CNTs electrodes was evaluated previously [18]. Since the diameters of nanotubes directly affect the microstructure on the surface of CNTs electrodes, here, we report the size-dependent voltammetric properties of CNTs electrodes. We found that electrodes made of nanotubes with smaller diameter exhibited better performance in voltammetric responses than that made of nanotubes with larger diameter. Besides, we found that electrodes made of nanotubes with mixed diameters exhibited the best voltammetric behaviors, and the voltammetric response was reversible.

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## 2. Experiments

Multi-walled carbon nanotubes (MWNTs) with different diameters were purchased from Helix Material Solutions Inc. The nanotubes were dispersed and functionalized by following protocol. First, 2 mg MWNTs was mixed with 40 ml Tween 20 (1% in water) and sonicated for 30 min in a water-bath sonicator. Then the mixture was centrifuged for 10 min at 2000 rpm and 30 ml upper supernatant was collected carefully without disturbing the bottom part (10 ml). This step eliminated most big aggregates of MWNTs. The collected supernatant was then filtered with a disc membrane filter (pore size 200 nm) and the MWNTs pellet on the membrane was re-suspended in 4 ml water by brief sonication. The temperature of water bath was then adjusted to 55–58 °C and the MWNTs resuspension was continually sonicated while 2 ml nitric acid (15 M) was added drop by drop. After that the mixture was further sonicated for 20 min and then incubated at 45 °C for 4 h under shaking. After incubation, MWNTs was collected again by filtering and washed with copious water to remove nitric acid and other oxidized impurities. Glass slides ( $0.6 \times 1$  cm) were cleaned by boiling in Piranha's solution (concentrated  $\text{H}_2\text{SO}_4$ : 30%  $\text{H}_2\text{O}_2$ , volume ratio 7:3) for 30 min and rinsed with copious water. Aqueous solution of MWNTs (0.4–0.6 mg/ml) was applied onto the glass slide and dried on a heat block (60 °C). This process was repeated

several times until the final thickness of the MWNTs film reached about 20  $\mu\text{m}$  (measured by SEM from side view).

The electrochemical studies were carried out in a typical three-electrode cell, using 1 M KCl solution containing 10 mM  $\text{Fe}(\text{CN})_6$ . Cyclic voltammetric measurements were performed using the MWNTs as working electrodes, a platinum wire as counter electrode and a SCE as a reference electrode. The data were recorded with an EG&G 273A Potentiostat/Galvanostat interfaced with a computer. All of the reagents are of analytic purity as supplied by Sigma–Aldrich, and deionized water obtained from a Millipore water system was used throughout. All electrochemical experiments were performed at room temperature ( $\sim 23$  °C).

## 3. Results and discussion

### 3.1. Characterization of the diameter of MWNTs and morphology of the electrodes

We prepared a set of electrodes with MWNTs of different diameters. Based on Fig. 1a, diameters of MWNTs range from 3 to 10, 10 to 20, 20 to 40 and 3 to 100 nm for electrodes 1#, 2#, 3# and A#, respectively. Diameter distributions were shown in Fig. 1b. The length of all the MWNTs ranged from 0.5 to 4  $\mu\text{m}$ .

The surface morphology of these MWNTs electrodes were shown in Fig. 2 and the zoomed images are shown

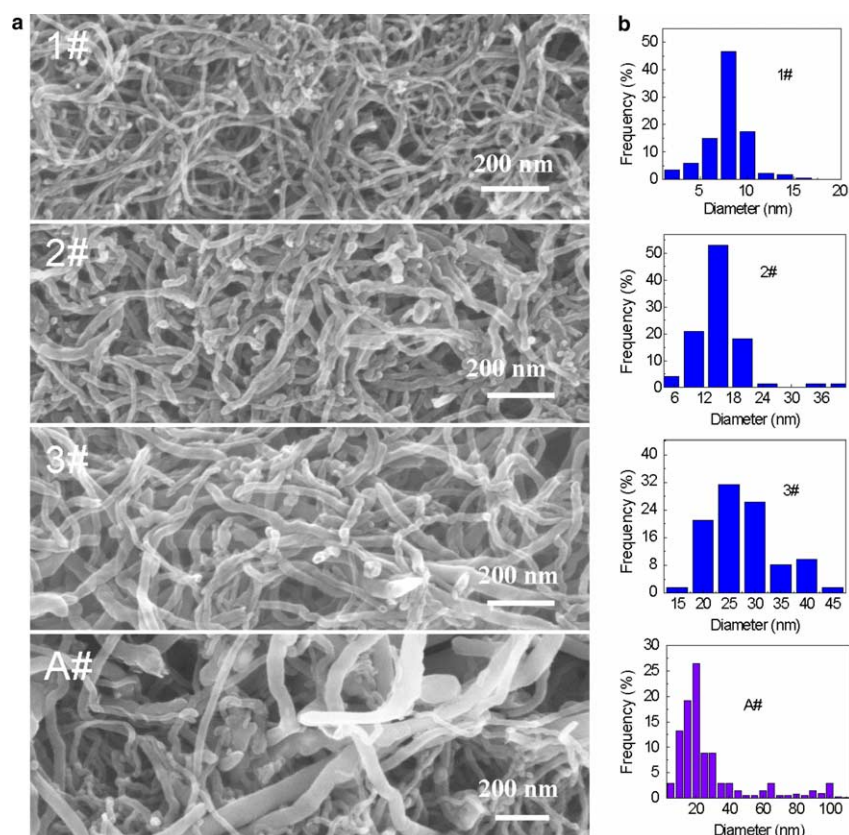


Fig. 1. (a) SEM images of the MWNTs electrodes and (b) the statistical diameter distributions by counting nanotubes from TEM images, indicating diameters of MWNTs range from 3 to 10, 10 to 20, 20 to 40 and 3 to 100 nm for electrode 1#, 2#, 3# and A#, respectively.

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