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Application of Carbon Nanotubes Modified Electrode in Pharmaceutical Analysis

Lingbo Qu[1,2] and Suling Yang[1]
[1]*Department of Chemistry, Zhengzhou University, Zhengzhou 450001,*
[2]*Chemistry and Chemical Engineering School, Henan University of Technology, Zhengzhou 450001,*
PR China

1. Introduction

The development of electrochemical sensors has attracted considerable attention as a low-cost method to the sensitive detection of a variety of pharmaceutical analytes. Since the discovery of carbon nanotubes (CNTs) in 1991 [1], research on CNTs has grown rapidly. In recent years, CNTs have also been used as electrode modified materials because CNTs offer unique advantages including enhanced electronic properties, a large edge plane/basal plane ratio, and electron transfer reactions [2]. Thus, CNTs-based sensors generally have higher sensitivities in a low concentration or in the complex matrix, lower limits of detection, and faster electron transfer kinetics than traditional carbon electrodes. Many factors need to be investigated in order to create an optimal CNTs-based sensor. Electrode performance can be influenced by the pretreatment of the nanotube, CNTs surface modification, the method of electrode attachment, and the addition of electron mediators. With the further development of CNTs and nanotechnology, studies on preparation, properties and application of CNTs-based modified electrodes have still been a hot topic attracting lots of researchers in the world. This article is presented on the application of CNTs modified electrode in different pharmaceutical analytes, which mainly includes the electrochemical studies on weak basic pharmaceuticals, weak acidic pharmaceuticals and other related small biological molecules. The physical and catalytic properties make CNTs ideal for use in sensors. Most notably, CNTs display high electrical conductivity, chemical stability, and mechanical strength.

2. CNTs modified electrode used in analysis of weak basic pharmaceuticals

Caffeine (3, 7-dihydro-1, 3, 7-trimethyl-1H-purine-2, 6-dione) and theophylline (3, 7-dihydro-1, 3-dimethyl-1H-purine-2, 6-dione), are two important active alkaloids that are widely distributed in beverages and plant products mainly including tea, coffee bean, cocoa and cola nuts. They are known to have many pharmacological effects, such as gastric acid secretion, diuretic, cardiac stimulant, and stimulant of central nervous system [3]. However, appropriate dosing is crucial because of the serious side adverse reactions in the presence of high concentrations of these compounds as the risk factors for asthma, kidney malfunction and cardiovascular diseases [4].

Nafion, a perfluorinated sulphonated cation exchanger with properties of excellent antifouling capacity, chemical inertness and high permeability to cations, has been extensively employed as an electrode modifier. CNTs can be homogeneously dispersed in Nafion solution because of the hydrophobic side chains and polar head groups of Nafion. Nafion/CNTs composite thin film-modified electrodes have their attractive effects in electroanalytical applications. Recently, we [5, 6] reported that Nafion/CNTs -modified electrode was made, and used as a sensor in the electrochemical determination of caffeine and theophylline. This sensor can ameliorate the problems of high overpotential, slow electrode reaction, and low sensitivity, which occurs at conventional electrodes.
Figure 1 displays the characterization of the Nafion/CNTs composite film on the glassy carbon electrode (GCE) by using scanning electron microscopy (SEM). It is obvious that the Nafion/CNTs composite film was uniformly coated on the electrode surface and formed a spaghetti-like porous reticular formation. The special surface morphology offered a much larger real surface area than the apparent geometric area.

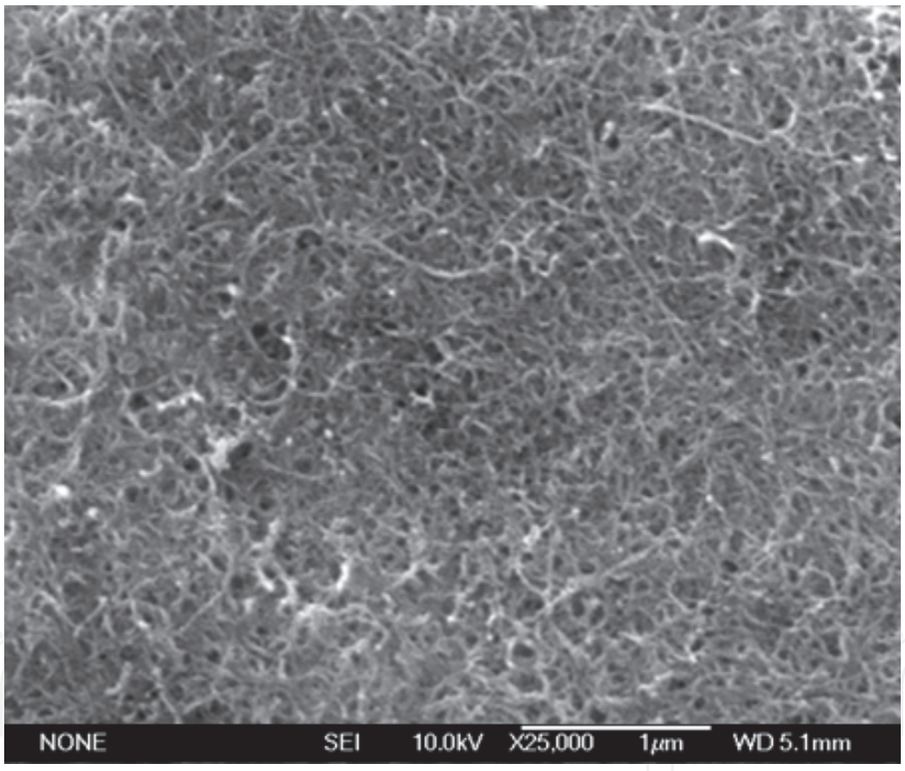

Fig. 1. SEM image of Nafion/CNTs composite film on glassy carbon electrode.

The experimental results demonstrated that caffeine and theophylline can be effectively accumulated at Nafion/CNTs composite film-modified electrode and produce a sensitive anodic peak in a 0.01 mol/L H_2SO_4 medium, respectively (Figure 2). Under the same conditions, no anodic peak of caffeine and theophyline was observed at the bare GCE. Especially at the Nafion/CNTs nanocomposite -modified electrode, the peak current was significantly higher than those at the CNTs/GCE or the Nafion/GCE. The oxidation process of caffeine and theophyline at Nafion/CNTs/GCE or Nafion/GCE is irreversible. Compared with the Nafion/GCE, the oxidation potential at the Nafion/CNTs/GCE was

negatively shifted. This phenomenon may be an evidence of catalytic effect of CNTs toward caffeine and theophyline oxidation. The reasons for the notable sensitivity of the determination at the Nafion/CNTs/GCE may be summarized as follows:(1) the Nafion/CNTs/GCE contains the cation exchanger of Nafion which has selective cation exchange enriched ability due to the electrostatic interaction. (2) CNTs display attractive characteristics, such as much larger specific surface area, excellent adsorptive ability and catalytic ability. Without a doubt, the synergetic functions of Nafion and CNTs make contributions to the higher current response [7-12].

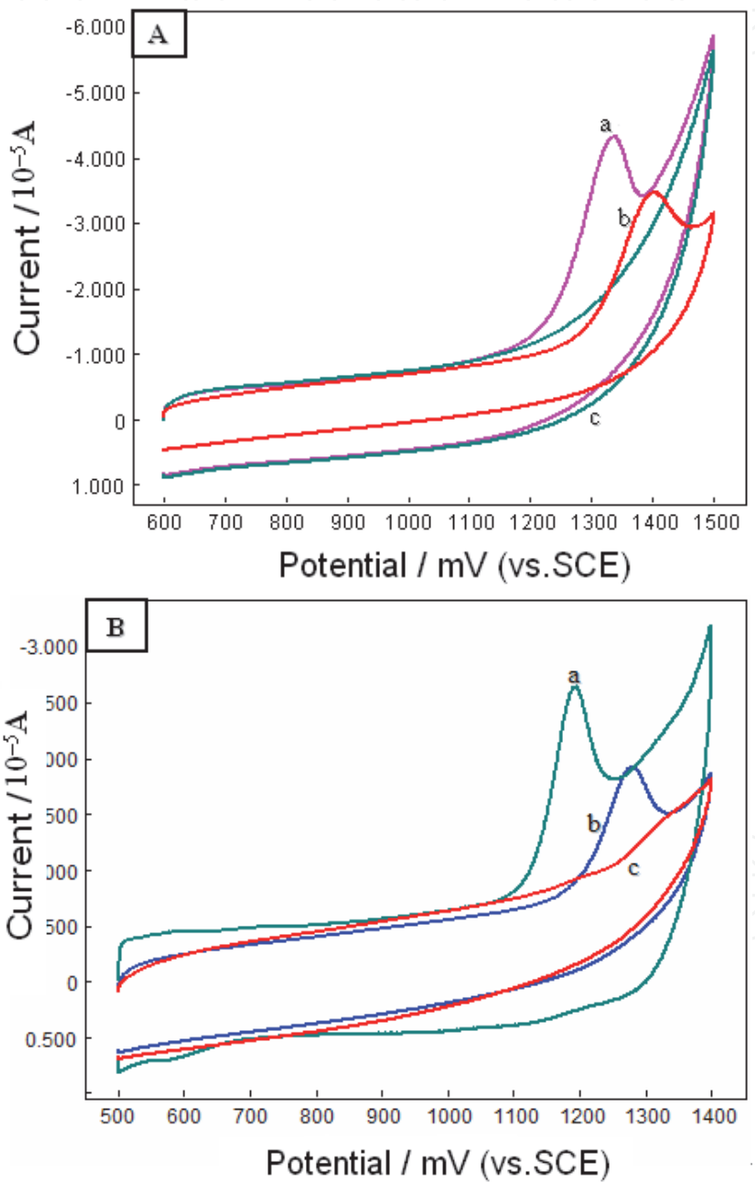

Fig. 2. Cyclic voltammograms of the Nafion/CNTs-modified GCE (a), Nafion-modified GCE (b), the bare GCE (c) in 0.01 mol/L H_2SO_4 medium containing: A. 3.0 ×10^{-5} mol/L caffeine; scan rate 80 mV/s; B. 2.0 × 10^{-5} mol/L theophyline; scan rate 100 mV/s.

Experiments revealed that the redox peak currents of rutin could be remarkably enhanced on AuNPs/en/CNTs/GCE, meaning good electrocatalytic activity for the oxidation of rutin. Figure 12 showed the cyclic voltammograms of rutin on different modified electrodes. Rutin didn't display any redox peaks at the bare GCE (a), which demonstrated the weaker adsorption and slower electrochemical reaction of rutin on the GCE surface. However, there were well-defined redox peaks on the en/CNTs/GCE (b), CNTs/GCE (c) and AuNPs/en/CNTs/GCE (d) in 0.1 mol/L phosphate buffer solution (pH 3.5). But the heights of the redox peaks were clearly higher in the case of AuNPs/en/CNTs/GCE than that of the redox peaks on the en/CNTs/GC or CNTs/GCE. The anodic (E_{pa}) and cathodic peak potentials (E_{pc}) were at about 487 mV and 432 mV (vs. SCE), respectively, and the ratio of i_{pa}/i_{pc} was about 1.2, which showed that the electrode reaction was almost reversible. Nano-gold and CNTs can enhance the electron-transfer rate and make more rutin participate in the electrochemical reaction due to their accumulation and catalytic ability.

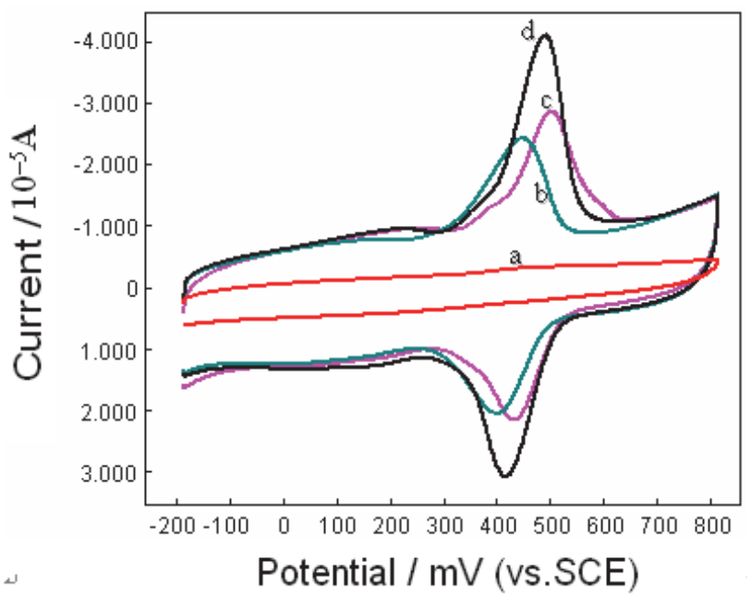

Fig. 12. Cyclic voltammograms of 8.0×10^{-5} mol/L rutin in 0.1 mol/L phosphate buffer (pH 3.5) on the different electrodes: the bare GCE (a), en/CNTs /GCE (b), CNTs/GCE (c) and AuNPs/en/CNTs/GCE (d); scan rate 100 mV/s.

Generally, as the electroactive substance, ascorbic acid (AA) always coexists in the Compound Rutin Tablets. The oxidation peak potential of AA is very close to that of rutin, which results in poor selectivity determination of AA or rutin in real samples on conventional electrodes. Therefore, it is essential to exploit more sensitive, selective and simple methods for the segregative determination of AA and rutin. Figure 13 demonstrated the cyclic voltammetry (CV) curves of 1.0×10^{-4} mol/L AA (a), the mixture of 2.0×10^{-5} mol/L rutin and 1.0×10^{-4} mol/L AA (b), 2.0×10^{-5} mol/L rutin (c) and without AA and rutin (d) in 0.1 mol/L phosphate buffer solution (pH 3.5) on AuNPs/en/CNTs/GCE. Figure 13 b showed two anodic peaks at around 186 mV and 487 mV, which were attributed to the oxidation of AA and rutin with a 301 mV separation of both peaks, indicating broad enough separation for the simultaneous electrochemical determinations of rutin and AA in the mixed solution.

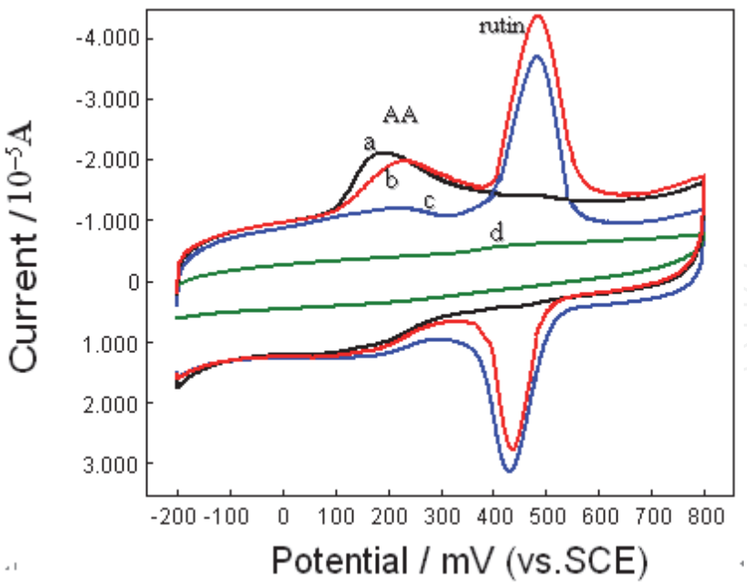

Fig. 13. Cyclic voltammograms of 1.0×10^{-4} mol/L AA (a), the mixed solution of 1.0×10^{-4} mol/L AA and 2.0×10^{-5} mol/L rutin (b), 2.0×10^{-5} mol/L rutin (c) and 0.1 mol/L phosphate buffer solution (pH 3.5) (d) on the AuNPs/en/CNTs/GCE; scan rate 100 mV/s.

Under optimized conditions, the anodic peak current was linear to the rutin concentration in the range of 4.8×10^{-8} mol/L − 9.6×10^{-7} mol/L. The regression equation was: $i_{pa} = 2.3728 C_{rutin} - 0.1782$ (i_{pa}: 10^{-5}A, C_{rutin}: μmol/L, $r = 0.9973$). The detection limit of 3.2×10^{-8} mol/L was obtained. Regeneration and reproducibility are two important characteristics for the modified electrode, which should be investigated. The same modified GCE was used for six times successive measurements of 2.0×10^{-5} mol/L rutin. After each measurement, the surface of the AuNPs/en/CNTs/GCE was regenerated by successively cycling between -200 mV and 800 mV (vs. SCE) in 0.1 mol/L phosphate buffer solution (pH3.5) for six cycles. The relative standard deviation (RSD) of the anodic peak current was 4.3%, which suggested good regeneration and reproducibility of the modified electrode.

This method was used for the determination of rutin in the Compound Rutin Tablets and Rutin Tablets. The contents of rutin in the Compound Rutin Tablets and Rutin Tablets were calculated to be 20.0 ± 0.87 mg and 19.4 ± 0.54 mg per tablet, respectively (the declared content of rutin was 20 mg per tablet). In order to test the accuracy of the proposed method, the conventional method of HPLC was employed to determine the contents of rutin in the Compound Rutin Tablets and Rutin Tablets (the contents of rutin in the Compound Rutin Tablets and Rutin Tablets were 18.9 ± 0.47 mg and 18.1 ± 0.57 mg per tablet, respectively). The quantitative results obtained by HPLC were in agreement with the data determined by the proposed electrochemical method, indicating that the method was selective and suitable for rutin determination in real samples.

6. Preparation of yttrium hexacyanoferrate/carbon nanotube/Nafion nanocomposite film-modified electrode: Application to the electrocatalytic oxidation of L-cysteine

L-cysteine (L-CySH), a sulfur-containing molecule, is one of the most important amino acids. It is widely present in many medicines, food and biological tissues, such as cysteine

protease, vasopressin and anti-diuretic hormone [40]. It plays a significant role in biological systems, playing a role in folding and defolding mechanisms [41]. An inadequate dietary intake of L-CySH may cause number of clinical problems, for instance, liver damage, skin lesions, and slowed growth [42]**.** Therefore, the sensitive detection of L-CySH is clinically significant. Nevertheless, some traditional methods, such as the chromatographic methods, are time-consuming, expensive, and require complicated preconcentration, multisolvent extraction and trained technicians. In contrast, electrochemical methods are characterized by their simplicity, high sensitivity, good stability, low-cost instrumentation, small scale and on-site monitoring [43]. However, although L-CySH is an electroactive compound, there are some drawbacks in electroanalysis, for example, large overpotential, low sensitivity and sluggish electron-transfer kinetics at conventional electrodes. These obstacles can result in oxide formation and fouling of the electrode surface [44]. To overcome these obstacles, we have successfully prepared YHCFNP, and the electrocatalytic oxidation and amperometric determination of L-CySH were accomplished at a modified electrode compose of a mixture of YHCFNP and CNTs [45].

As can be seen in figure 14, when YHCFNP and carbon nanotube were mixed together, they were uniformly dispersed. In this case, CNTs can be used as the carrier and entanglement (diameter: 10 – 20 nm, length: 1–2 μm) to fix YHCFNP on the electrode surface.

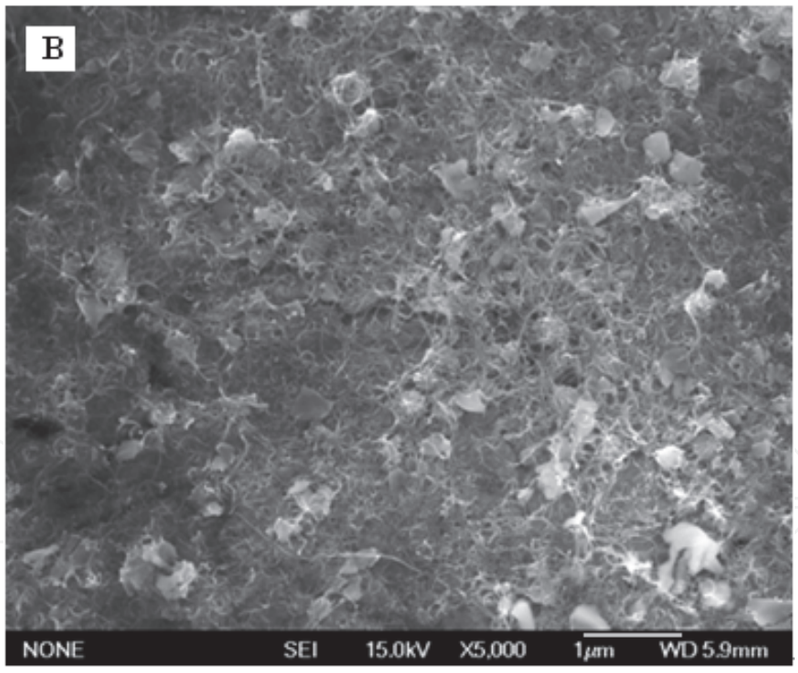

Fig. 14. Typical SEM images of the mixed YHCFNP and CNTs

Figure 15 shows the cyclic voltammograms of different electrodes with and without 0.5 mmol/L L-CySH in 0.1 mol/L PBS (pH 6.82). As figure 15A reveals, at the YHCFNP/CNTs/Nafion-modified GCE, there is a pair of well-defined redox peaks at 152 mV and 250 mV at a scan rate of 20 mV/s without L-cysteine. Under the same conditions, there was no redox peak at the CNTs/Nafion-modified GCE and bare GCE.

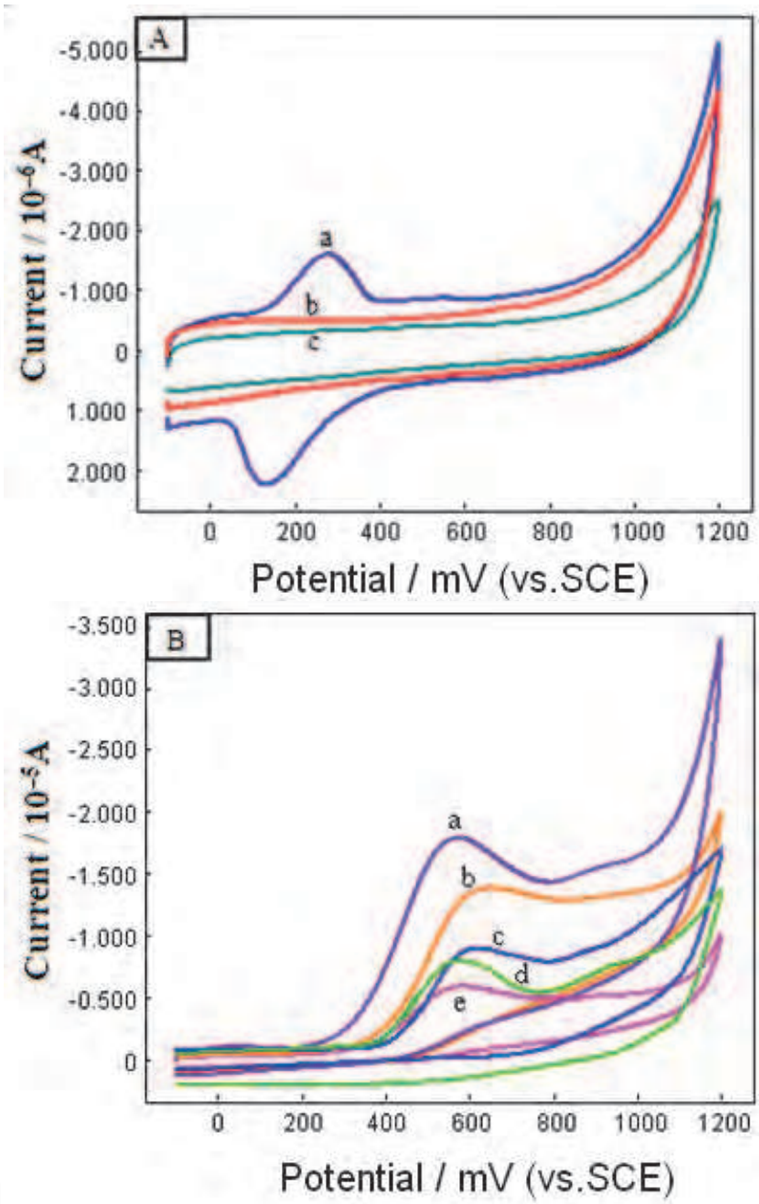

Fig. 15. A. Cyclic voltammograms of different electrodes in 0.1 mol/L PBS (pH 6.82) without L-cysteine: YHCFNP/CNTs/Nafion-modified GCE (a), CNTs/Nafion-modified GCE (b), GCE (c) ; B. Cyclic voltammograms of different electrodes in mol/L PBS (pH 6.82) with 0.5 m mol/L L-cysteine: YHCFNP/CNTs/Nafion-modified GCE (a), CNTs/Nafion-modified GCE (b), CNTs-modified GCE (c), YHCFNP-modified GCE (d), GCE (e). Scan rate 20 mV/s.

Figure 15 B depicts the cyclic voltammograms of different electrodes with the addition of 0.5 m mol/L L-CySH in 0.1 mol/L PBS (pH 6.82). L-cysteine produced a weak anodic peak at the bare GCE (c), which demonstrates the slower electrochemical reaction of L-cysteine at the GCE surface. This phenomenon may be related to electrode fouling caused by the deposition of this compound and its oxidation products on the electrode surface. At the CNTs-modified GCE (c) , there was a well-defined anodic peak , and the peak

current was clearly higher than that at the bare GCE. Indeed, in a number of cases, the L-cysteine molecules under study can interact with carbon nanotube in a way that a well-polished ''traditional'' carbon electrode cannot. This phenomenon is evidence of the catalytic effect of CNTs toward L-cysteine oxidation. In most cases, the electrocatalytic activity of carbon nanotube is attributed to edge plane like-sites/defects, which may occur at the ends, and along the tube axis [46]. Recently it has been revealed that additionally, metallic impurities remaining from the fabrication processes can be the origin for certain analytes [47–49]. Because of the excellent evenly dispersing capacity of Nafion for CNTs, a stronger anodic peak of L-CySH was shown at the CNTs/Nafion-modified GCE (b). At the YHCFNP-modified GCE (d), a well-defined anodic peak appeared. The peak current was obviously higher than that at the bare GCE, which demonstrated that the YHCFNP-modified GCE can improve the electrochemical reaction of L-cysteine on the electrode surface. In addition, a well-defined anodic peak appeared at the YHCFNP/CNTs/Nafion-modified GCE (a), and the height of the anodic peak was clearly higher than that at the CNTs/Nafion-modified GCE. The anodic potential (E_{pa}) was about 570 mV. In contrast, the anodic potential at the CNTs/Nafion-modified GCE was 642 mV, indicating a negative shift of about 72 mV. The experimental results indicate that the electrooxidation of L-CySH is remarkably improved by the YHCFNP/CNTs/Nafion-modified GCE, which may result from the high dispersion of YHCFNP/CNTs nanocomposite with high surface area and good electronic properties.

Under the optimum experimental conditions, the electrochemical response to L-cysteine at the YHCFNP/CNTs/Nafion-modified GCE was fast (within 4 s). Linear calibration plots were obtained over the range of 0.20–11.4 μmol/L with a low detection limit of 0.16 μmol/L. The YHCFNP/CNTs/Nafion-modified GCE exhibited several advantages, such as high stability and good resistance against interference by ascorbic acid and other oxidizable amino acids.

7. Concluding remarks

Nowadays, the study of CNTs modified electrode is the forefront subject that offers enormous possibilities in pharmaceutical analysis. The facility of the preparation and functionalization of CNTs makes them very useful for the development of modified electrodes with specific detection of medicine molecules, as well as for the fabrication of third generation sensors (no mediator is needed). As far as the modified electrode is concerned, the CNTs coated and polymer embedded electrodes are more widely used than the CNTs paste electrode and CNTs intercalated electrode in the determination of pharmaceutical analysis owing to the different dispersants. Thus, to explore and use the materials as dispersants with good and green friendly properties is an important field. Moreover, CNTs intermingled with other nonmaterials (such as nano Au particles, nano YHCF, *etc*) were modified on conventional electrodes. These nanocomposites provide a synergic effect which results in the improvement in the response property of modified electrodes. So, CNTs hybrid with other nanomaterials used as modified material was also an important part. In addition, further research on the mechanism of the electrochemical reaction between medicine and modified electrode is a very important aspect in relation to CNTs modified electrode. Although CNTs modified electrode used in pharmaceutical analysis is receiving increasing interest for sensor construction in recent years, the study should involve the combination of nanomaterials, analysis and life sciences.

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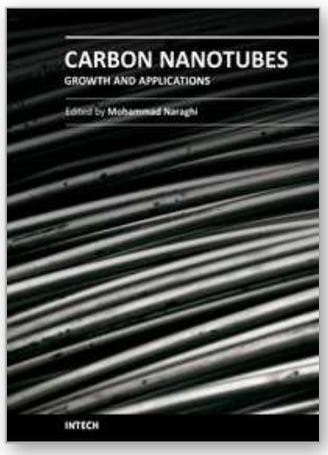

Carbon Nanotubes - Growth and Applications

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Carbon Nanotubes are among the strongest, toughest, and most stiff materials found on earth. Moreover, they have remarkable electrical and thermal properties, which make them suitable for many applications including nanocomposites, electronics, and chemical detection devices. This book is the effort of many scientists and researchers all over the world to bring an anthology of recent developments in the field of nanotechnology and more specifically CNTs. In this book you will find:

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