



# Synthesis of bismuth nickelate nanorods and electrochemical detection of tartaric acid using nanorods modified electrode



L.Z. Pei\*, T. Wei, N. Lin, H. Zhang

Key Lab of Materials Science and Processing of Anhui Province, School of Materials Science and Engineering, Anhui University of Technology, Ma'anshan, Anhui 243002, PR China

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## ABSTRACT

Single crystalline bismuth nickelate nanorods with cubic  $\text{Bi}_{12}\text{NiO}_{19}$  phase and flat tips have been successfully synthesized by a facile hydrothermal route without any additives. Electron microscopy observations show that the length and diameter of the bismuth nickelate nanorods are 1–3  $\mu\text{m}$  and 30–100 nm, respectively. The formation, length and diameter of the nanorods depend on the hydrothermal temperature and duration time. The formation of the bismuth nickelate nanorods are explained according to the nucleation and crystalline growth process. The bismuth nickelate nanorods are applied as the glassy carbon electrode (GCE) materials for the detection of tartaric acid in KCl solution. The roles of scan rate, electrolyte species and tartaric acid concentration on the electrochemical responses of tartaric acid have been investigated. The obtained linear concentration calibration plot shows a low detection limit of 0.52  $\mu\text{M}$ , wide linear range of 0.001–2 mM and correlation coefficient of 0.996. The bismuth nickelate nanorods modified GCE shows good reproducibility and stability.

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## 1. Introduction

The determination of the organic acids is a challenging and interesting topic in analytical biochemistry because organic acids are widely present in beverages, body fluids, food and pharmacology. Tartaric acid belongs to the group of hydrocarboxylic acid, which is widely used in the fields of pharmacology, chemistry and food industries [1,2]. Therefore, it is essential to detect tartaric acid by a rapid, accurate and sensitive method.

Some methods and electrodes have been used for the determination of tartaric acid. A disk-shaped copper electrode was reported to simultaneously detect tartaric acid, ascorbic acid glucose, sucrose and fructose by co-electroosmotic capillary electrophoresis with amperometric method [3]. Tartaric acid could be detected within 14 min with relative standard deviation (R.S.D.) of less than 5%. The detection limit was 0.71  $\mu\text{M}$  calculated based on three times ratio of signal to noise and linear range was 0.01–0.50 mM. Chen et al. [4] also reported that the tartaric acid, citric acid, malonic acid, oxalic acid, aspartic acid and glutamic acid could be simultaneously detected by capillary electrophoresis using a copper disk electrode. The linear range for the determination of tartaric acid was

0.008–1 mM. The stopped-flow chemiluminescence spectrometry method was used for the determination of tartaric acid by the oxidation with periodate. The linear range was 0.005–0.05 mM, detection limit was 1.5  $\mu\text{M}$  and R.S.D. was less than about 2% [5]. The glassy carbon electrode (GCE) modified with  $\beta$ -Cyclodextrin, carbon nanotubes and methylene blue was designed for the electrochemical detection of tartaric acid [6]. The electrochemical detection performance of tartaric acid at the copper sulfide modified GCE had been analyzed by the electrochemical cyclic voltammetry (CV) method. The detection limit and linear range were 0.32  $\mu\text{M}$  and 0.001–2 mM, respectively [7]. Although some methods have been used for the determination of tartaric acid, these methods still take the disadvantages of expensive apparatus, complex measurement process and low sensitivity. Therefore, it is of important significance to explore novel electrode materials, sensitive method for the detection of tartaric acid.

Ternary metal oxide one-dimensional (1D) nanoscale materials show great application potential in electrochemical sensors for the determination of organic acids [8–12]. For example, copper germanate nanowires modified GCE was used to detect tartaric acid in neutral solution with the detection limit of 7.7  $\mu\text{M}$  [13]. Among ternary oxide 1D nanoscale materials, bismuth nickelate 1D nanoscale materials may be promising GCE modified materials in electrochemical sensors due to large surface area and good

\* Corresponding author.

E-mail addresses: [lzpei@ahut.edu.cn](mailto:lzpei@ahut.edu.cn), [lzpei1977@163.com](mailto:lzpei1977@163.com) (L.Z. Pei).

electrochemical performance. Generally, bulk bismuth nickelate could be prepared by high temperature solid method [14–16]. Du et al. [17] reported the synthesis of three-dimensional (3D) NiO/Bi<sub>7.47</sub>Ni<sub>0.53</sub>O<sub>11.73</sub> (BiNiO) microspheres by a mixed solvothermal process using urea. However, to date, no bismuth nickelate 1D nanoscale materials, such as nanorods are reported. Therefore, it is desirable to explore the synthesis of bismuth nickelate nanorods for the possible applications in electrochemical sensors.

Hydrothermal method takes the advantages of simple hydrothermal process, low cost, good repeatability, high crystallinity and purity exhibiting great application promising for the synthesis of 1D nanoscale materials [18–25]. In this paper, single crystalline bismuth nickelate nanorods have been synthesized by a simple hydrothermal process without additives. The bismuth nickelate nanorods have been characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM) and high-resolution TEM (HRTEM) to analyze the morphology and structure of the nanorods. The morphology and phase evolution of the bismuth nickelate products have been investigated by analyzing the SEM images and XRD patterns of the products obtained from different hydrothermal factors. The bismuth nickelate nanorods are applied as the GCE modified materials for the detection of tartaric acid. The electrochemical behaviors of tartaric acid at the nanorods modified GCE have been investigated by cyclic voltammetric (CV) method. The performance of the bismuth nickelate nanorods modified GCE, such as tartaric acid concentration linear range, detection limit and correlation coefficient have been discussed. The bismuth nickelate nanorods exhibit excellent electrocatalytic activities toward tartaric acid and application potential in electrochemical sensors.

## 2. Experimental

### 2.1. Materials

Sodium bismuthate (NaBiO<sub>3</sub>·2H<sub>2</sub>O, AR grade) was supplied by Aladdin Reagent Co., Ltd. of P. R. China. nickel acetate (C<sub>4</sub>H<sub>6</sub>O<sub>4</sub>Ni·4H<sub>2</sub>O, AR grade) and tartaric acid (AR grade) were supplied by Sinopharm Chemical Reagent Co., Ltd. of P. R. China. All chemicals were used without further purification during the experimental process.

### 2.2. Methods

Bismuth nickelate nanorods were synthesized by the hydrothermal method without additives. In a typical experiment, 0.16 g nickel acetate and 0.2 g sodium bismuthate were dissolved into 60 mL deionized water sequentially with stirring. Afterward, the mixture was transferred into a Teflon-lined stainless steel autoclave with a filling capacity of 60%. The autoclave was sealed and underwent hydrothermal treatment at 80–180 °C for 0.5–24 h. Then the autoclave was cooled down naturally. The resulting brown bismuth nickelate precipitates were collected by the centrifugation, washed using deionized water and ethanol for several times and the products were dried at 60 °C in air. Finally, brown bismuth nickelate nanorods powders were obtained.

### 2.3. Characterization

The phase composition and crystal structure of the products were identified by XRD using a Bruker AXS D8 X-ray diffractometer with a graphite monochromatized Cu K $\alpha$  radiation ( $\lambda = 1.5406 \text{ \AA}$ ) source. The samples were scanned at a scan rate of  $0.05 \text{ }^\circ\text{s}^{-1}$  in  $2\theta$  range of 20–80°. The morphologies of the products were examined using SEM, TEM and HRTEM. SEM observation was performed using

a nova nanoSEM FEI 430 SEM operating at an acceleration voltage of 20 kV. For TEM and HRTEM observations, the bismuth nickelate nanorods samples were prepared by putting several drops of solution with bismuth nickelate nanorods onto a standard copper grid with a porous carbon film after the bismuth nickelate nanorods were dispersed in deionized water and treated for about 10 min using ultrasonic bath. TEM and HRTEM observations were performed using a JEOL JEM-2100 TEM with the accelerating voltage of 200 kV and a GATAN digital photography system.

### 2.4. Preparation of bismuth nickelate nanorods modified GCE and electrochemical detection of tartaric acid

Bismuth nickelate nanorods suspension was prepared by dispersing 10 mg bismuth nickelate nanorods into 10 mL N,N-dimethylformamide (DMF) solvent with sufficient ultrasonication for 1 h. GCE (3 mm in diameter) was polished to a mirror-like surface with metallographic sand paper and 0.05  $\mu\text{m}$  alumina suspension. The treated GCE was cleaned in an ultrasonic cleaner with alcohol and water, respectively. Bismuth nickelate nanorods modified GCE was prepared by casting 10  $\mu\text{L}$  nanorods suspension onto the surface of the GCE and dried under an infrared lamp. Electrochemical measurements were recorded using a model CHI604D electrochemical analyzer (Shanghai CH Instrument Company, PR China). All CVs were obtained in a three-electrode system. A platinum plate was used as the counter electrode, saturated calomel electrode (SCE) was applied as the reference electrode and bismuth nickelate nanorods modified GCE was used as the working electrode. Electrochemical impedance spectroscopy (EIS) measurement was performed using Fe(CN)<sub>6</sub><sup>3-</sup> as a probe with an amplitude of 10 mV and frequency ranging from 3500 Hz to 50 Hz. All potentials were recorded with respect to SCE. CVs were recorded in three-electrode cell between the potential range of  $-1.0 \text{ V}$  and  $+1.0 \text{ V}$  at a scan rate of  $50 \text{ mV s}^{-1}$  in the mixed solution of 0.1 M KCl and tartaric acid with different concentrations. All experiments were conducted at room temperature.

## 3. Results and discussion

### 3.1. Characterization of bismuth nickelate nanorods

The typical XRD pattern of the bismuth nickelate products obtained from 180 °C for 24 h is shown in Fig. 1. All diffraction peaks of

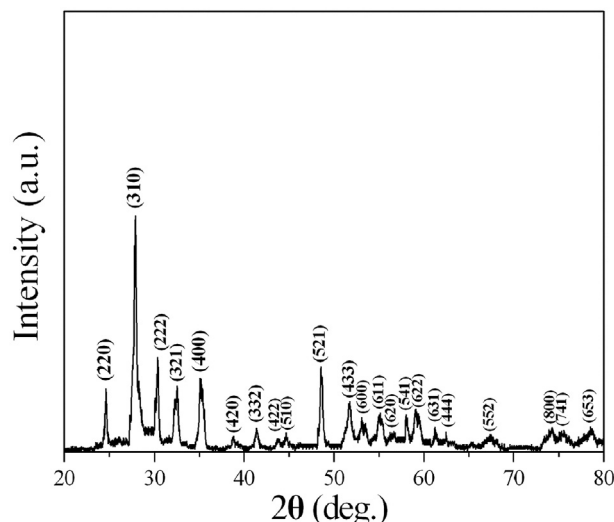


Fig. 1. XRD pattern of the bismuth nickelate nanorods obtained from the hydrothermal conditions of 180 °C for 24 h.

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