



Direct growth of ternary copper nickel cobalt oxide nanowires as binder-free electrode on carbon cloth for nonenzymatic glucose sensing

Hadi Mirzaei^a, Ali Akbar Nasiri^a, Rahim Mohamadee^a, Hajar Yaghoobi^b, Mehrdad Khatami^c, Omid Azizi^d, Mohammad Ali Zaimy^e, Hakim Azizi^{f,*}

^a Department of Biotechnology, Zabol University of Medical Sciences, Zabol, Iran

^b Cellular and Molecular Research Center, Shahrekord University of Medical Sciences, Shahrekord, Iran

^c School of Medicine, Bam university of Medical Sciences, Bam, Iran

^d Department of Medical Basic Sciences, Torbat Heydarieh University of Medical Sciences, Torbat Heydarieh, Iran

^e Department of Medical Genetics, Tehran University of Medical Sciences, Tehran, Iran

^f Department of Medical Parasitology, School of Medicine, Zabol University of Medical Sciences, Zabol, Iran

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ABSTRACT

A new binder free electrode based on ternary copper nickel cobalt oxide nanowires grown on the carbon cloth (CuNiCoO₄ NWs@ carbon cloth) was prepared and characterized by field emission scanning electron microscopy (FE-SEM), x-ray diffraction (XRD), Energy-dispersive X-ray spectroscopy (EDX), and cyclic voltammetry (CV). The prepared electrocatalyst was directly used for electrochemical sensing of glucose without using enzyme. The effect of different parameters such as potential scan rate, switching potential, and glucose concentration on the electrochemical oxidation of glucose was investigated. The results showed that such an electrode presents excellent catalytic activity toward the oxidation of glucose in aqueous alkaline solution. Under optimum conditions, the potential application of the electrode was evaluated by applying it to the analytical determination of glucose concentration. The results revealed that the electrocatalytic current increased linearly with the glucose concentration in the range from 0.02 to 1.4 mM with a low detection limit of 6.5 μM and good sensitivity of as high as 1782 μA mM⁻¹ cm⁻². Selectivity investigations demonstrate that the CuNiCoO₄ NWs@CC electrode could be used for selective detection of glucose in the presence of interfering species. Real sample analysis shows reasonable RSD values implying negligible matrix effect in determination of glucose in human serum samples.

1. Introduction

Rapid, sensitive, selective and reliable glucose sensing is important for wide variety of industries and sciences such as food industry, biotechnology, environmental, and clinical monitoring [1]. So far, various glucose sensors have been established based on optical, acoustic, electronic, fluorescent, electrochemical methods and et cetera [2]. Between these glucose sensors, electrochemical sensors are of high great importance because of their high sensitivity, specific selectivity and low cost [3]. Since Clark's and Lyon's work on enzyme glucose biosensor in 1962 [4], determination of glucose attracted particular attention. Commercialized sensors for glucose monitoring utilized enzymes such as glucose oxidase, which adheres particularly to β-D-glucose and then it catalyzes the glucose oxidation to hydrogen peroxide and D-glucono-δ-lactone. The transduce signal is originates from the oxidation of hydrogen peroxide that is produced in the presence of sensing element

(enzyme) [1, 5, 6]. Although, these sensors show high sensitivity and selectivity toward glucose oxidation [5, 7, 8], but, they usually suffered from short lifetime and their efficiency are simply affected by the changes in pH values, humidity of the media, temperature, the enzyme immobilization techniques and the attendance of toxic chemicals during measurements [9–11].

Recently, electrochemical methods have attracted increasing attention for determination of glucose and other analytes [12–15]. However, Non-enzymatic glucose sensors of noble metals such as Pd [16], Au [17] and Pt [18] or their corresponding amalgams [19, 20] widely utilized for glucose measurement. However, these electrode materials have high costs which limits their wide applications in various field of sciences. Hence, many attempts have been done to design new and cost-effective catalyst materials. In this regard, one component-based transition metal oxides such as Co₃O₄ [21], NiO [1, 22], Cu₂O [23] or binary metal oxides and hydroxides such as CuO_x-CoO_x

* Corresponding author.

E-mail address: hakimazizi190@yahoo.com (H. Azizi).

[24], $\text{Cu}_2\text{O}/\text{NiO}_x$ [25], $\text{Ni}_x\text{Co}_{2x}(\text{OH})_{6x}$ [26], NiCo_2O_4 [27, 28] have been attracted an extensive attention.

Commonly, in a typical electrochemical nonenzymatic glucose sensor, the electrocatalysts are produced in the form of nanoparticles, and then immobilized on conductive substrates by definite usually non-conductive and electrochemically inactive polymeric binders. The existence of the polymeric binders in the electrochemical nonenzymatic glucose sensor not only enhances the series resistance values but also blocks the active sites of the catalyst and hinders the electrolyte ions diffusion to the surface of the catalyst, eventually results in a significantly reduced electrocatalytic activity and poor performance of the sensor. However, using binder-free electrocatalysts grew directly on the surface of conductive substrates could overwhelmed such problems [1, 29–31]. However, to the best of our knowledge, there is no report on the preparation of ternary metal oxides for glucose sensing in alkaline media.

In this work, CuNiCoO_4 nanowires constructed on carbon cloth electrode (CuNiCoO_4 NWs@CC) as nonenzymatic glucose sensor were proposed for the first time. The ternary metal oxide nanowires uniformly covered the carbon cloth surface and formed three-dimensional heterostructures. Due to the presence of more active catalytic sites, the resulting nanowires showed a high performance for the non-enzymatic determination of glucose. The electrochemical and electrocatalytic behaviors of the ternary metal oxide nanowires toward the oxidation of glucose were evaluated by cyclic voltammetry and hydrodynamic amperometry methods. The results showed that this new ternary metal oxide could be an excellent candidate for electrocatalytic oxidation of glucose in alkaline media.

2. Experimental

2.1. Materials

The copper nitrate trihydrate ($\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$), cobalt nitrate hexahydrate ($\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$), nickel nitrate hexahydrate ($\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$), ammonium fluoride (NH_4F) and urea ($\text{CO}(\text{NH}_2)_2$) were purchased from Merck Chemicals Company. All used aqueous solutions were made up in deionized (DI) water. All the chemicals used in the experiments were of analytical grade without further purification.

2.2. Synthesis of CuNiCoO_4 NWs@CC

The growth of CuNiCoO_4 NWs arrays on CC was performed using a facile hydrothermal synthesis method that combined with a calcination procedure. Before deposition process, a piece of commercial CC substrates was ultrasonically cleaned in acetone, deionized water and ethanol, respectively and dried in an oven. Then, a reaction solution including 1 mM of $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$, 1 mM of $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, 1 mM of

$\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, 6 mM of ammonium fluoride (NH_4F) and 15 mM urea ($\text{CO}(\text{NH}_2)_2$) in distilled water was prepared under constant magnetic stirring for 30 min. After putting a piece of cleaned CC ($1 \text{ cm} \times 1 \text{ cm}$), a portion of 70 mL of the prepared solution was transferred into 100 mL Teflon-lined stainless steel autoclave and the temperature was maintained at 120°C for 6 h. After the autoclave spontaneously cooled to room temperature, in order to remove residual reactants and free debris, the CC that covered with mixed metal (Cu, Ni, Co) carbonate hydroxide precursor film was carefully rinsed with de-ionized water and absolute ethanol in succession. Then, the prepared carbonate hydroxide precursor was dried using vacuum at 60°C for 12 h. Finally, the precursor sample was put in a quartz tube furnace and thermal annealing was performed at 350°C (2°C min^{-1}) in argon atmosphere for 2 h, which a full transform of precursor to hierarchical structure of Cu-Ni-Co ternary oxide (CuNiCoO_4) supported on CC was occurred in this step.

2.3. Materials characterizations

The crystallographic structure of the prepared samples was investigated by X-ray diffraction (XRD) equipped with $\text{Cu K}\alpha$ radiation (λ : 1.78901 \AA). The microstructure and chemical composition of the samples were determined by using a field-emission scanning electron microscope (FE-SEM) that equipped with an energy-dispersive X-ray spectrometer (EDX) (TESCAN). All of the electrochemical measurements were performed on Autolab PGSTAT101 at the room temperature.

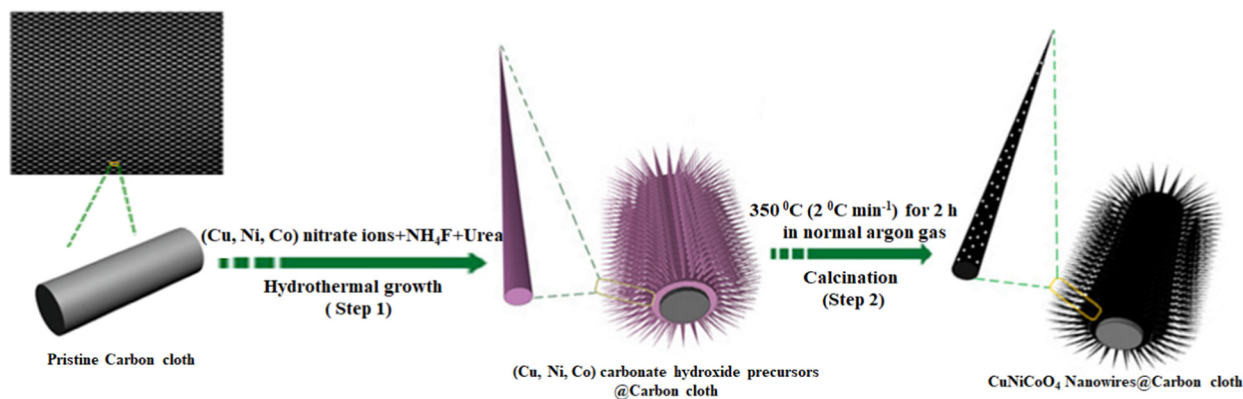
2.4. Electrochemical studies

Electrochemical performance of the as-synthesized hierarchical nanostructured electrode was studied in aqueous solution of NaOH (0.1 M) with three electrode cell configuration which CuNiCoO_4 NWs@CC was directly used as a working electrode, a platinum plate as a counter electrode, and a Ag/AgCl (3.5 M KCl) as a reference electrode. Cyclic voltammetry (CV) tests were done to study the oxidation behavior of glucose at the CuNiCoO_4 NWs@CC. Amperometric response of glucose sensor was measured in a stirred medium. Before adding glucose solutions and concentration measurements, the background current was allowed to fall to a steady value.

3. Results and discussion

3.1. Characterization of CuNiCoO_4 NWs@CC

The nano-architecture active materials were prepared using a simple two-step synthesis strategy (Scheme 1) containing directly growth of mixed Cu-Ni-Co precursors on CC by hydrothermal reaction



Scheme 1. The schematic overview of CuNiCoO_4 NWs@carbon cloth fabrication.

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