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One-step electrochemical exfoliation of nanoparticles-assembled NiO nanosheets for non-enzymatic glucose biosensor

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ABSTRACT

NiO nanosheets were successfully exfoliated from the paired metal Ni wires by one-step electrochemical process in blank NaOH electrolyte. The nanosheets were composed of agglomerated nanoparticles, with the thickness of the nanoscale, nano/micro size, and abundant lacerated edges. The NiO nanosheets modified glass carbon electrode demonstrated excellent electrocatalytic activities and performance in sensing glucose with high sensitivity (838.09 μA mM^{-1} cm^{-2}), wide linear concentration range (500 nM to 2.31 mM), low detection limit (0.145 μM), good anti-interference, and reproducibility, which was further applied to detect glucose in a serum sample.

1. Introduction

The earth-abundant transition metal oxide NiO has become one of the ideal materials to construct glucose biosensors, due to its low price, well selectivity, high catalytic rate, and not easy to adsorb intermediates and Cl^- ions [1,2]. However, its poor conductivity and high contact resistance between structures limited the electronic transmission in sensors. Typically, the metal oxide NiO is composite with low-dimensional or precious metal nanomaterials to improve the performance [3–5]. Another strategy to enhance the performance of electrochemical NiO based glucose sensors is to directly fabricate low-dimensional NiO nano-structures (nanoflake arrays [6], nanofibers [7], nanosheets [8,9] and so on).

Herein, we reported a facile electrochemical route to exfoliate NiO nanosheets from metal Ni wires at room temperature with simple devices and operation, no use of toxic reagents and Ni salts precursors. Due to significantly increasing electrocatalytic activities of electrode materials for glucose molecules, the NiO nanosheets modified glass carbon electrode (NiO nanosheets/GCE) demonstrated excellent sensitivity, low detection limit and anti-interference in sensing of glucose.

2. Experimental

The one-step electrochemical exfoliation of NiO nanosheets and construction of glucose sensor were illustrated in Fig. 1(A). Briefly, 5 V alternating voltage at 50 Hz was applied with a pair of nickel wires immersed in continuously stirred 4 M NaOH solution for 30 min. Subsequently, the products in electrolyte were collected by centrifuging and rinsed with ultrapure water and absolute ethanol. The detailed preparation, characterization, electrode modification and electrochemical measurements of samples are provided in Supplementary information.

3. Results and discussion

XRD pattern of electrochemically exfoliated products is shown in Fig. 1(B), a series of diffraction peaks can be assigned to the (1 1 1), (2 0 0), (2 2 0), (3 1 1) and (2 2 2) lattice planes of face-centered cubic bunsenite NiO (JCPDS No. 89-7130). Importantly, no other impurity phases observed indicated high purity due to all products directly derived from pure Ni wires. SEM images in Fig. 1(C) and inset show the prepared NiO nanosheets with the thickness of the nanoscale, the nano/micro size, and abundant lacerated edges. The nanosheets were composed of massive agglomerated nanoparticles in Fig. 1(D), which is actually a quite common phenomenon in the various self-assembled NiO nanostructures [10]. In contrast, the commercial bulk NiO revealed an aggregated structure containing numerous discrete micro or nano/particles as shown in Fig. S1.

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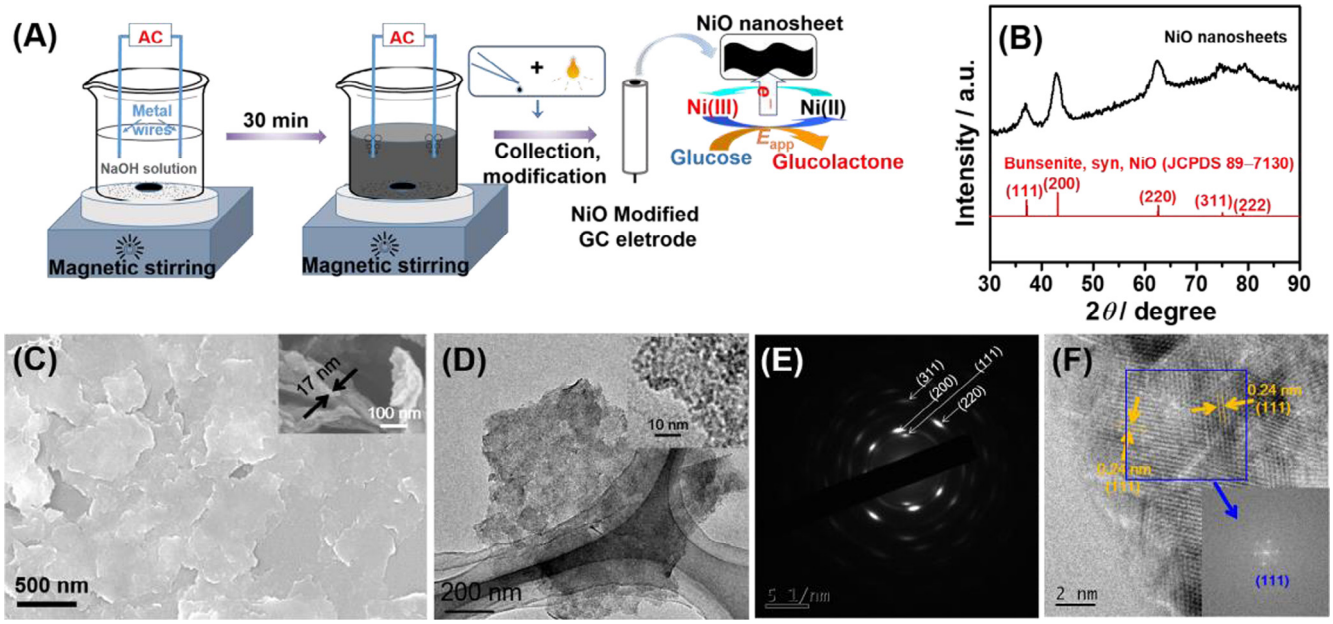

Fig. 1. (A) Illustration for electrochemical exfoliation and construction of glucose sensor. (B) XRD pattern, (C) SEM images, (D, F) TEM images, (E) SAED pattern of the NiO nanosheets.

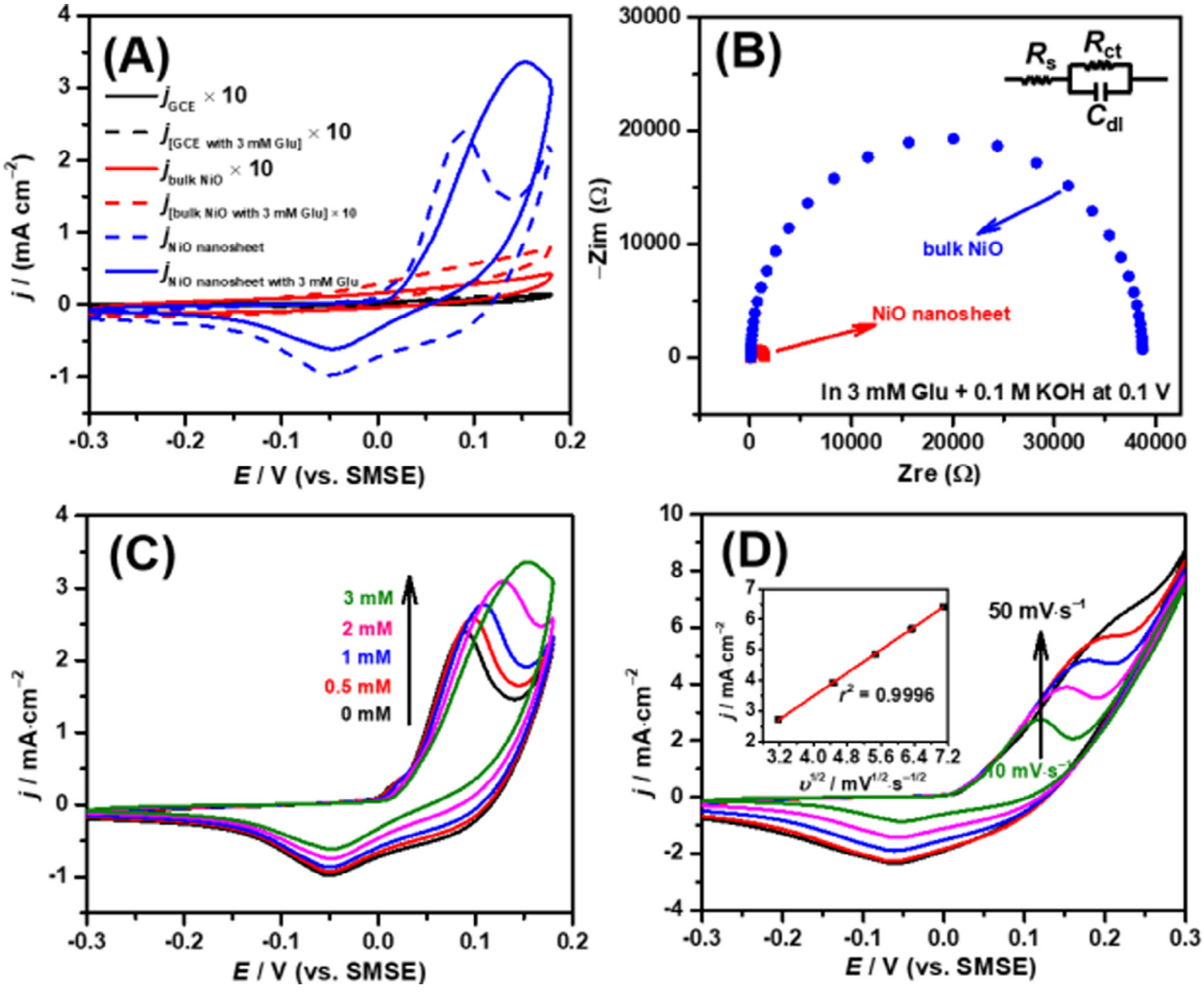

Fig. 2. (A) CVs at 10 mV s^{-1} in 0.1 M KOH on different electrodes. The *j* of GCE and bulk NiO/GCE was enlarged ten times. (B) Nyquist plots of EIS with bulk NiO and NiO nanosheet. CVs (C) at 10 mV s^{-1} with different concentrations of glucose and (D) at different scan rates with 1 mM glucose in 0.1 M KOH on the NiO nanosheets/GCE electrode.