



# In-situ fabrication of 3D flower-like $\text{NH}_4\text{NiPO}_4$ on Ni foam without nickel salts added for high sensitive nonenzymatic glucose detection

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## ABSTRACT

The 3D flower-like  $\text{NH}_4\text{NiPO}_4$  was successfully fabricated on nickel foam via one-step hydrothermal route without nickel salts added. In our design, nickel ions were originated from the acid corrosion of nickel foam, which decreased the concentration of nickel ion in the solution and effectively control the environmental pollution. The 3D flowers-like  $\text{NH}_4\text{NiPO}_4$  was arranged on the Ni foam, and the 3D flower-like structures were assembled by sheet-like crystals with an average thickness of 380 nm. Furthermore, the sheet-like crystals are also stacked by many layers of nanoflakes. The 3D flower-like  $\text{NH}_4\text{NiPO}_4$  modified Ni foam can be used as a binder-free electrode for non-enzymatic glucose detection. And the electrode exhibits excellent electrochemical properties with high sensitivity ( $6135 \mu\text{A mM}^{-1} \text{cm}^{-2}$  and  $2205 \mu\text{A mM}^{-1} \text{cm}^{-2}$ ), wide linear range (covered from 0.001–1 mM and 2–5 mM) and a low detection limit of 0.015 (3S/N) at an applied bias of 0.5 V vs. Ag/AgCl.

## 1. Introduction

Ni-based materials (NiO, NiS and  $\text{Ni}(\text{OH})_2$ , et al.) have been widely used as non-enzymatic glucose sensors [1–4] due to its low cost and excellent electrochemical performance. However, the application of glucose detection is limited by their narrow linear range and low sensitivity. Hence, it is necessary to search for another material to enhance electro-catalytic activity as enzyme-free glucose sensor.

Recently,  $\text{NH}_4\text{NiPO}_4$  has received increasing attention due to its excellent energy storage properties as supercapacitor electrode material [5,6]. However, the application of  $\text{NH}_4\text{NiPO}_4$  for enzyme-free glucose sensor remains unexplored. So we try to evaluate the electrochemical activity of  $\text{NH}_4\text{NiPO}_4$  as enzyme-free glucose sensor.

Moreover, 2D micro/nanostructure, especially sheet-like crystal stacked by many layers of nanoflakes [7,8], shows a great surface area and high conductivity. Also, building blocks into a three-dimensional (3D) architecture can greatly decrease re-stack and condense caused by their high surface energy and interlayer van der Waals attractions, which combines the advantages of both microstructures and nanostructures [9]. Furthermore, the in-situ grown materials directly on the matrix is beneficial to increase the tightness of intimate interface and decrease contact resistance of different components [10,11] compared with traditional strategies [12–16]. Inspired by the above-mentioned concepts, in-situ construction of 3D architectures on the substrate can

be regarded as an important approach to improve their electro-chemistry performance.

Herein, we proposed a facile one-step low-temperature hydrothermal route to synthesize  $\text{NH}_4\text{NiPO}_4/\text{Ni}$  foam electrode without nickel salts added. In our design, nickel ions were originated from the acid corrosion of nickel foam, which decreased the concentration of nickel ion in the solution and control the environmental pollution effectively. The sheet-like  $\text{NH}_4\text{NiPO}_4$  crystals on nickel foam assemble as three-dimensional (3D) flowerlike structures. And the 3D flower-like  $\text{NH}_4\text{NiPO}_4/\text{Ni}$  foam electrode exhibits excellent electrochemical properties with high sensitivity for binder-free electrode as glucose sensor.

## 2. Materials and methods

### 2.1. Preparation of samples

In a typical procedure, the nickel foam ( $1 \times 1 \text{ cm}$ ) was firstly cleaned using dilute hydrochloric acid, acetone, absolute ethanol and deionized water, respectively. Then, 0.1 mL concentrated nitric acid, 0.004 mol diammonium hydrogen phosphate and 3 g urea were mixed with 50 mL deionized water. After that, the Ni foam was put into a Teflon-lined stainless steel autoclave filled with the mixed solution above mentioned. The autoclave was sealed and maintained at  $120^\circ\text{C}$  for 12 h and subsequently cooled to room temperature naturally.

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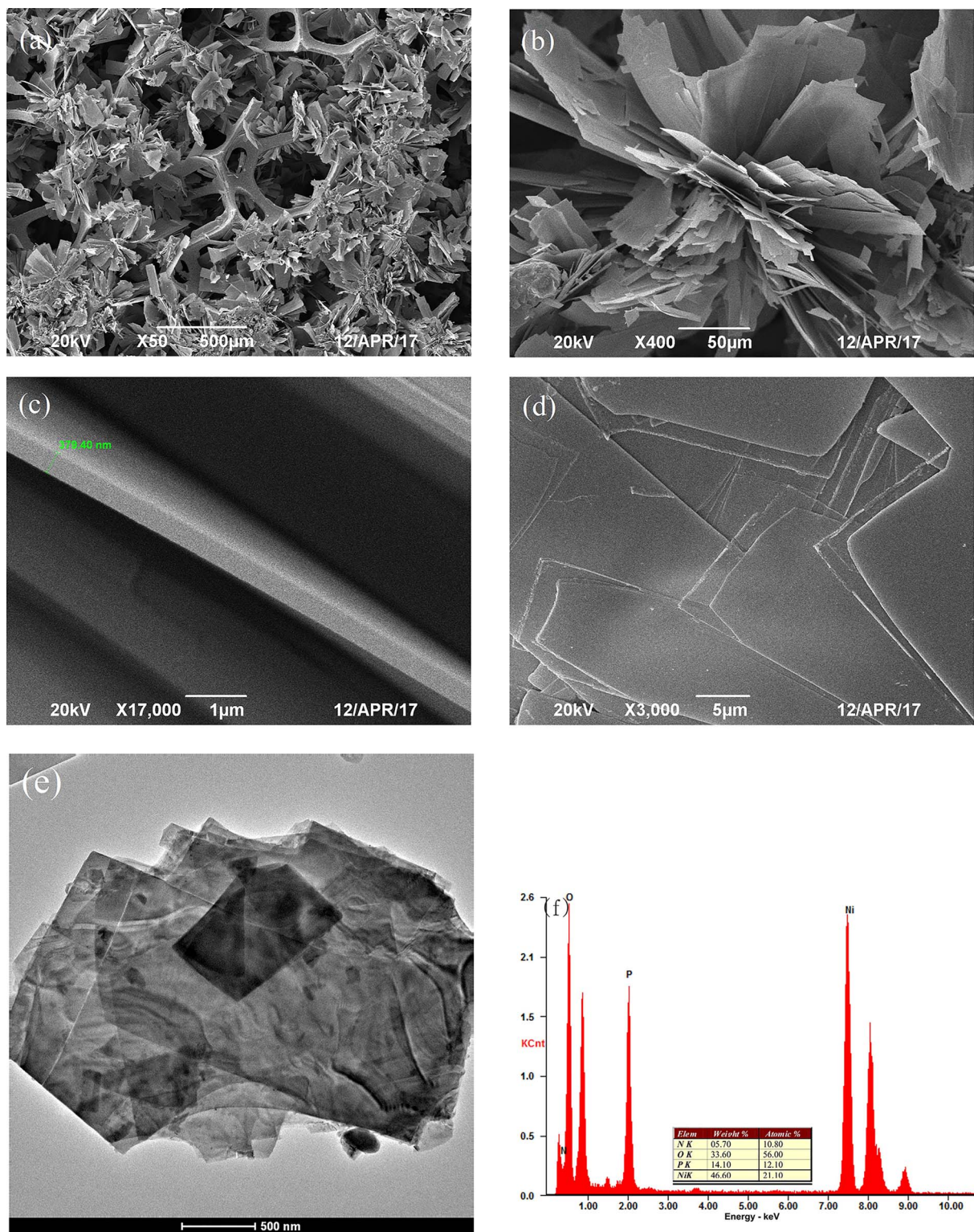


Fig. 1. SEM images (a–d), TEM images (e) and corresponding EDAX spectrum (f) of  $\text{NH}_4\text{NiPO}_4$  on the nickel foam.

Finally, the product was rinsed with deionized water and dried at  $60^\circ\text{C}$  in air.

## 2.2. Characterizations

The X-ray diffraction (XRD) patterns of the 3D flowerlike ammonium nickel phosphate/Nickel foam electrodes were examined by a DX-2700 diffractometer with a  $\text{Cu K}\alpha$  radiation source ( $\lambda = 0.15418\text{ nm}$ ).

The chemical composition and element chemical states of the samples were determined by X-ray photoelectron spectroscopy ESCALAB 250Xi (XPS, USA). The morphologies and microstructures of the products were investigated by means of scanning electron microscopy (JEOL JSM-6490LV). Transmission electron microscopy (TEM) images and energy dispersive x-ray analysis (EDAX) were taken on a FEI Tecnai F20 TEM.

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