

## Cathodic deposition of binary nickel-cobalt hydroxide for non-enzymatic glucose sensing

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### ABSTRACT

Binary Ni-Co hydroxide is directly grown on screen-printed carbon electrodes (SPCEs) through cathodic deposition for detecting glucose. The material has been identified to consist of nickel-cobalt hydroxide (denoted as (Ni-Co)(OH)<sub>2</sub>) by X-ray photoelectron spectroscopic analysis. From cyclic voltammetric (CV) and chronoamperometric (CA) results, (Ni-Co)(OH)<sub>2</sub> shows a more explicit signal than Co(OH)<sub>2</sub> and a less positive detecting potential than Ni(OH)<sub>2</sub> for glucose oxidation. Such synergistic performances suggest the atomic scale mixing of Ni and Co ions in the hydroxide matrix. The amperometric detection of this non-enzymatic glucose sensing shows a linear calibration curve up to 3700 μM with a sensitivity of 122.45 μA/(mM cm<sup>2</sup>) ( $R^2 = 0.989$ ). The interference responses of 25 μM ascorbic acid (AA), uric acid (UA), and dopamine (DA) were 10.76, 14.29 and 1.41% of the signal of 0.2 mM glucose, respectively. The binary (Ni-Co)(OH)<sub>2</sub> exhibits a low detecting potential, a high signal-to-noise ratio, and a wide calibration-curve range for the non-enzymatic glucose sensing.

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## 1. Introduction

Reliable and fast methods for monitoring glucose have been of great interests for vital applications of electrochemistry [1,2]. Although the glucose meter for the blood sugar determination is the most important application of glucose sensing, the development of suitable chemical sensors for monitoring the glucose in food and drink products, such as measuring the quantity of sugars, the nutritional value of raw materials, final products and technological processes, is also being actively pursued by academic and industrial researchers [3,4].

In order to gain excellent sensitivity and high selectivity of the blood sugar meter, enzymatic glucose sensors, such as oxygen-mediated electrode, artificial mediator type, and direct electron-transfer type, have been widely developed [5]. However, they generally show serious issues in stability, simplicity, reproducibility, and the oxygen limitation. Accordingly, a number of noble metal electrode materials, e.g., platinum [6], gold [7] and alloys [8], were explored for the non-enzymatic glucose detection. However, high cost and poor selectivity of noble metals hindered the practical applications in various fields although direct oxidation of glucose can be enhanced [9].

Recently, new materials for the non-enzymatic glucose sensing have been proposed [10] and several oxides, e.g., CuO [11], MnO<sub>2</sub> [12], NiO<sub>x</sub> [13], and CoO<sub>x</sub> [14], were investigated for such applications. Among them, NiO<sub>x</sub> and CoO<sub>x</sub> receive much attention due to their electrocatalytic properties and chemical stability [15]. Meanwhile, glucose oxidation is mainly catalyzed by Ni(OH)<sub>x</sub> or Co(OH)<sub>x</sub> under higher oxidation states in the alkaline media [16,17]. Although Ni oxy-hydroxides generally show a higher sensitivity for detecting glucose than Co oxy-hydroxides, the anodic peak potential shift and stability still remain big problems for the applications [3,18]. In this work, a binary nickel-cobalt hydroxide simply prepared by means of cathodic deposition is demonstrated to show a lower sensing potential, a higher sensitivity, and a wider calibration-curve range for the non-enzymatic glucose sensor in comparison with pure Ni and Co hydroxides.

## 2. Experimental

All hydroxides were cathodically deposited onto screen-printed carbon electrodes (SPCEs) at 5 mA/cm<sup>2</sup> for 100 s from the precursor baths at 70 °C. For depositing Ni(OH)<sub>2</sub> (or Co(OH)<sub>2</sub>), the deposition bath contains 0.13 mM NaNO<sub>3</sub> and 0.07 mM NiCl<sub>2</sub>·6H<sub>2</sub>O (or CoCl<sub>2</sub>·6H<sub>2</sub>O). For binary hydroxide deposition, the baths contain 0.2 mM NaNO<sub>3</sub>, 0.066 mM NiCl<sub>2</sub>·6H<sub>2</sub>O, and 0.033 mM CoCl<sub>2</sub>·6H<sub>2</sub>O. After deposition, the electrodes were rinsed with de-ionized water and dried at room temperature overnight.

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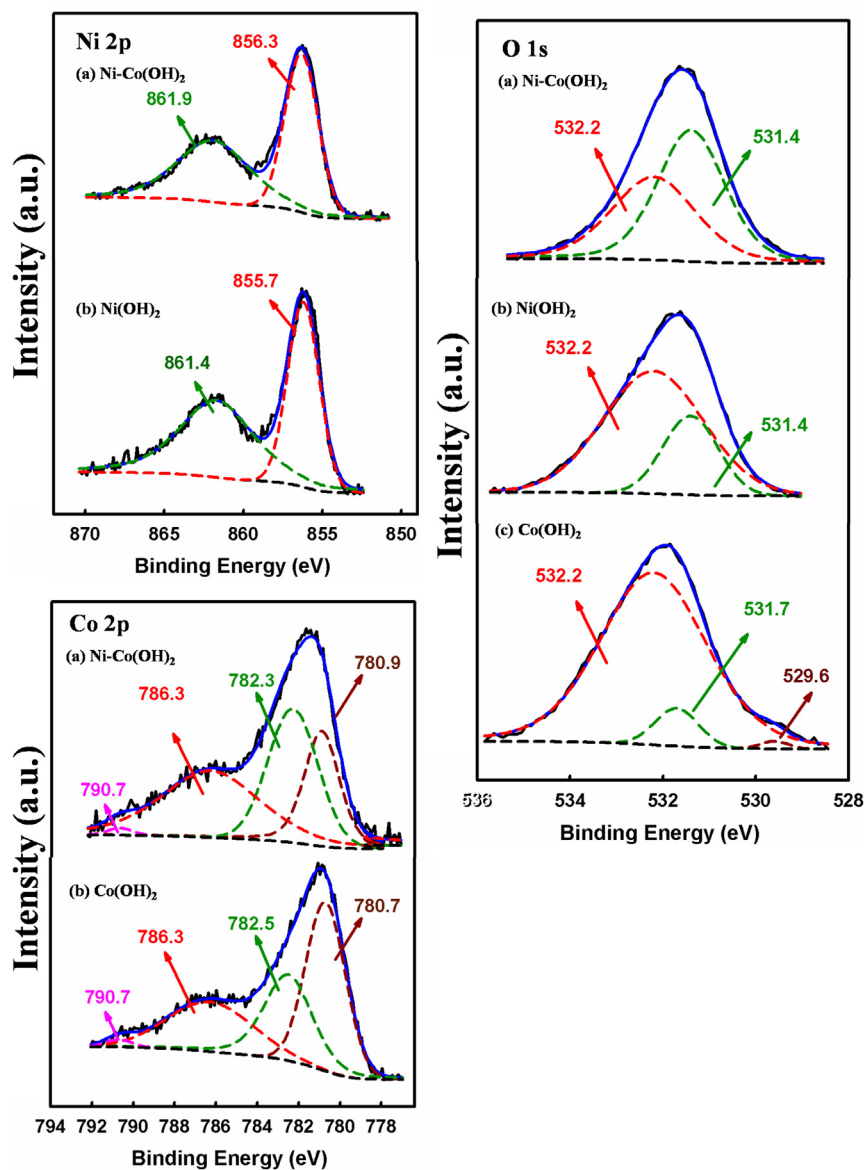


Fig. 1. XPS core-level spectra of Ni 2p, Co 2p and O 1s of (a) and (e) (Ni-Co)(OH)<sub>2</sub>, (b) and (g) Ni(OH)<sub>2</sub>, and (d) and (f) Co(OH)<sub>2</sub>.

The cathodic deposition and electrochemical analyses were performed in a three-compartment cell. The counter and reference electrodes were a platinum mesh (99.95%) and Ag/AgCl (Argenthal, 3 M KCl, 0.207 V vs. SHE at 25 °C), respectively. The geometric area of commercially available SPCEs (Zensor R&D) is 0.196 cm<sup>2</sup>. Chronoamperometric (CA) and cyclic voltammetric (CV) analyses were measured by an electrochemical analyzer (CHI633c, CH Instruments Inc.) in 0.5 M NaOH without or with 0.1 M PBS (phosphate buffer solution). X-ray photoelectron spectroscopy (XPS) was measured by a Kratos Axis Ultra DLD (Kratos Analytical, USA) with an Al K $\alpha$  ( $h\nu = 1486.69$  eV) X-ray source. All solutions were prepared with deionized water produced by a reagent water system (Milli-Q SP, Japan) at 18 M $\Omega$  cm. All reagents without further purification were from Merck. Solution temperature was maintained at specified temperatures with an accuracy of 0.1 °C by a water thermostat (Haake DC3 and K20).

### 3. Results and discussion

Chemical environments of all materials prepared in this work were characterized by XPS and the XPS core-level spectra of Ni 2p,

Co 2p, and O 1s for all samples are shown in Fig. 1. Note that both Ni 2p and Co 2p exhibit spin-orbit splitting into 2p<sub>1/2</sub> and 2p<sub>3/2</sub> which qualitatively contain the same information [19]. Thus, only the higher-intensity peaks (i.e., Ni 2p<sub>3/2</sub> and Co 2p<sub>3/2</sub>) are subjected to curve-fitting in this work. Curves (a) and (b) show the Ni 2p<sub>3/2</sub> spectra with the fitting curves for Ni-Co and Ni hydroxides, respectively. The main peaks on curves (a) and (b) are respectively centered at 856.3 and 856.2 eV, reasonably assigned to Ni(OH)<sub>2</sub> (856.2 ± 0.2 eV) [20]. Curves (c) and (d) respectively show the Co 2p<sub>3/2</sub> spectra and their fitting data for binary Ni-Co and Co hydroxides, which can be decomposed into four constituents. The four constituents for Co(OH)<sub>2</sub> are centered at 780.7, 782.5, 786.3, and 790.7 eV which are exactly the same as those reported in the literature [19]. The peaks for the binary hydroxide are located at the same positions while the distribution of these constituents is slightly shifted, which may be attributed to the different neighboring cations in the hydroxide matrix. Moreover, the presence of a peak centered at 782.5 eV and the broad satellite peak at 786.3 eV has been assigned to the formation of cobalt hydroxide [19]. The above results suggest that Ni and Co ions in the hydroxide matrix are under the atomic scale mixing, leading to the synergistic performances in glucose sensing

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