



Development of selective and sensitive bicarbonate chemical sensor based on wet-chemically prepared CuO-ZnO nanorods



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ABSTRACT

We have prepared calcined copper oxide doped zinc oxide nanorods (CuO-ZnO NRs) by a facile wet-chemical method using reducing agents in alkaline medium. The doped NRs were totally characterized by UV/vis, FT-IR, X-ray photoelectron, energy-dispersive X-ray spectroscopy, X-ray powder diffraction, and field-emission scanning electron microscopy. The NRs were deposited on flat silver electrode (AgE) to result in a sensor that has a fast response to selective bicarbonate in buffer system. Features including high sensitivity, lower-detection limit, reliability, reproducibility, ease of integration, long-term stability, selective, and enhanced electrochemical performances were investigated in detail. The calibration plot is linear ($r^2 = 0.99$) over the large concentration range (1.0 nM to 1.0 mM). The sensitivity and detection limit are calculated as $\sim 1.667 \mu\text{A cm}^{-2} \mu\text{M}^{-2}$ and $0.89 \pm 0.02 \text{ nM}$ (at a signal-to-noise-ratio, SNR of 3) respectively. Finally, the efficiency of the proposed chemi-sensors can be applied and effectively utilized for the detection of bicarbonate in various environmental real water samples with acceptable and reasonable results.

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1. Introduction

A chemical sensor is a sensor that produces an electric signal proportional to the concentration of chemical or biochemical analytes. These chemi-sensors use chemical as well as physical principles in their operation. The sensor signal is significantly enhanced in the presence of nanomaterials or functional materials whether doped or undoped fabricated onto the sensor surfaces in chemical or biological systems. Doped nanomaterials have attracted a wide interest due to their unique properties and potential application in chemi-sensor fabrication [1,2]. Semiconductor material has been also recognized as a promising host nano-material for transition metal oxides at room conditions. It is revealed a stable morphological structure and composed of a number of irregular phases with geometrically coordinated metals and oxide atoms, piled alternately along the three dimensional axes [3]. Transition metals codoped in semiconductor nano-materials have

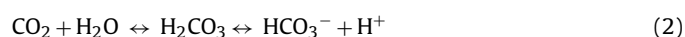
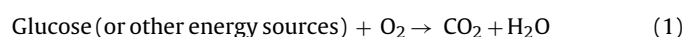
concerned insightful research effort for its exceptional and outstanding optical, structural, electrical, photocatalytic properties, and versatile applications [4]. Last decade, an extensive development has been executed on the research leading of transition metal oxides incorporated ZnO materials actuated by both fundamental sciences and prospective advanced technologies [5–7]. The doped semiconductor nanostructures exhibit promising uses as field consequence transistors [8], UV photo-detectors [9,10], gas sensors [11], field emission electron sources [12], nanomaterials [13,14], nanoscale power generators [15], and many other functional devices [16]. Transition metal doped nanostructure is also an effective method to regulate the energy level in surface states of ZnO, which can further progress by the changes in doping concentrations of host-guest materials. Chemical sensing expedition has been utilized with the metal oxide nanostructures for the detection of various chemicals such as ethanol, phenyl hydrazine, methanol, hydrazine, chloroform, dichloromethane, acetone, and ethanol which are not environmental friendly. The sensing mechanism with doped metal oxides thin films utilized mainly the properties of porous film formed by the physi-sorption and chemisorptions methods. The chemical detection is based on the current changes of the fabrication thin films caused by the chemical components of the reacting system in aqueous medium [17–20]. The main effort is focused on detecting the minimum quantity aqueous

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chemical necessary for the fabrication doped sensors for electrochemical investigation. Nanomaterials offer many opportunities of tuning the chemical sensing properties.

In physiological systems, carbon dioxide is obviously linked with bicarbonate (HCO_3^-) via carbonic anhydrases, and numerous biological processes are dependent upon a mechanism for sensing the level of HCO_3^- or carbon dioxide, and/or pH. In human body, the discovery that soluble adenylyl cyclase is directly regulated by bicarbonate (HCO_3^-) provided a link between $\text{HCO}_3^-/\text{CO}_2/\text{pH}$ chemi-sensing and signalling through the widely used second messenger cyclic adenosine monophosphate. This review summarizes the evidence that bicarbonate-regulated adenylyl cyclase, and additional, subsequently identified bicarbonate-regulate nucleotidyl cyclases, function as evolutionarily conserved $\text{HCO}_3^-/\text{pH}/\text{CO}_2$ chemi-sensors in a wide variety of physiological systems [21]. In physiological system, carbon dioxide and water are the major end products of energy producing pathways in living organisms according to Eq. (1). As such, in non-photosynthetic organisms, CO_2 and water represent the most fundamental catabolites.



In unicellular organisms, carbon dioxide gas can simply diffuse away, but once multi-cellular organisms evolved, they had to devise methods for safely dealing with carbon dioxide. In solution, CO_2 combines with water to form carbonic acid (H_2CO_3), which dissociates to liberate a proton and a bicarbonate ion (HCO_3^-) (Eq. (2)). Bicarbonate, carbon dioxide and pH equilibrate on their own within minutes, but in biological systems, equilibrium is reached nearly instantaneously due to the ubiquitous presence of carbonic anhydrases [22,23]. This equilibrium is used to buffer pH inside cells and in intercellular fluids; for example, intracellular pH is regulated via interplay between carbon dioxide diffusion, and bicarbonate and proton transporters and/or exchangers. In mammals, and terrestrial vertebrates in general, this equilibrium is tightly controlled in two ways; the kidneys regulate the bicarbonate concentration and the breathing frequency determines the concentration of carbon dioxide. Each of these processes requires a “sensor” i.e., an exquisitely sensitive and rapid way to measure the precise concentration of bicarbonate and/or carbon-dioxide and/or pH and elicit an appropriate response. Many other physiological processes, in addition to diuresis and breathing rate regulation, are modulated by bicarbonate and/or carbon dioxide and/or pH (i.e., sperm activation, blood flow, aqueous humour in the eye and cerebrospinal fluid formation), and they also require a $\text{HCO}_3^-/\text{carbon-dioxide}/\text{pH}$ sensor. For many years, the effects of carbon dioxide and pH had been ascribed to undefined chemo-receptors, and the effects of bicarbonate were traditionally thought to be mediated by changes in cellular pH. In 2000, researcher demonstrated that HCO_3^- directly modulates the activity of soluble adenylyl cyclase, a novel form of the enzyme generating the ubiquitous second messenger, cyclic adenosine monophosphate [24–26], revealing that physiological $\text{HCO}_3^-/\text{carbon dioxide}/\text{pH}$ could be sensed via second messenger signalling. In physiological systems, HCO_3^- , carbon dioxide, and pH are intimately linked via carbonic anhydrases and a variety of biological processes, in mammals and throughout evolution, depend upon a $\text{HCO}_3^-/\text{carbon-dioxide}/\text{pH}$ chemosensor. Bicarbonate-regulated adenylyl cyclase, which links intracellular HCO_3^- , carbon-dioxide, and/or pH levels with cyclic adenosine monophosphate signal transduction, serves as the $\text{HCO}_3^-/\text{carbon-dioxide}/\text{pH}$ chemi-sensor in at least a subset of these processes. The future will reveal whether other $\text{HCO}_3^-/\text{carbon-dioxide}/\text{pH}$ chemi-sensing functions are also mediated by adenylyl cyclase. Bicarbonate regulation is observed in other mammalian nucleotidyl cyclases and in adenylyl cyclases across evolution implying that

cyclic nucleotide signalling is an evolutionarily conserved mechanism for $\text{HCO}_3^-/\text{carbon-dioxide}/\text{pH}$ chemi-sensing.

In the present study, it is employed the wet-chemical technique to prepare CuO doped ZnO semiconductor nanomaterials with nearly controlled rod-shape structure, which revealed a continuous morphological advancement in nanostructure materials and potential applications. With most of the existing works focused on undoped ZnO, there have been more and more attention dedicated to explore the doped counterparts. For semiconductor nanomaterials, doping is an influential application to conform the optical and electrical properties, expediting the development of many electronic and opto-electronic devices. Semiconductor nanostructures CuO-ZnO NRs allow very sensitive transduction of the liquid/surface interactions into a change in the electrochemical properties. The possibility is to form a variety of structural morphologies suggests various prospects of tuning the chemical sensing properties. CuO-ZnO NR is fabricated by a simple and efficient chemical sensors consisting on a side-polished flat silver electrode surface; and measured the chemical sensing performance of bicarbonate in phosphate buffer system at room conditions. To best of our knowledge, this is the first report for detection of bicarbonate with prepared CuO-ZnO NRs using simple and reliable $I-V$ technique in short response time.

2. Experimental

2.1. Materials and methods

Zinc chloride, copper chloride, sodium bicarbonate, butyl carbitol acetate, ethyl acetate, ammonia solution (25.0%), and all other chemicals were in analytical grade and purchased from Sigma-Aldrich Company (<http://www.sigmaaldrich.com>). The λ_{max} (366.0 nm) of calcined CuO-ZnO NRs was executed using UV/visible spectroscopy Lambda-950, Perkin Elmer, Germany (<http://www.perkinelmer.com>). FT-IR spectra of CuO-ZnO NRs were performed on a spectrum-100 FT-IR spectrophotometer in the mid-IR range purchased from Bruker (<http://www.bruker.com>). The XPS measurement of CuO-ZnO NRs was measured on a Thermo Scientific K-Alpha KA1066 spectrometer (<http://www.thermoscientific.com>). A monochromatic Al K α 1 X-ray radiation source was used as excitation sources, where beam-spot size was kept in 300.0 μm . The spectra were recorded in the fixed analyzer transmission mode, where pass energy was kept at 200.0 eV. The scanning of the spectra was performed at pressures less 10^{-8} Torr. Morphology, size, and structure of calcined CuO-ZnO NRs were recorded on FESEM instrument from JEOL (JSM-7600F, Japan; <http://www.jeol.com/>). The powder X-ray diffraction (XRD) patterns of CuO-ZnO NRs were recorded by X-ray diffractometer from PANalytical diffractometer (<http://www.panalytical.com>) equipped with Cu K α 1 radiation ($\lambda = 1.5406 \text{ nm}$) using a generator voltage of 45.0 kV and a generator current of 40.0 mA were applied for the determination. Raman spectrometer was used to measure the Raman shift of calcined CuO-ZnO NRs using radiation source (Ar^+ laser line, λ ; 513.4 nm), which was purchased from Perkin Elmer (Raman station 400, Perkin Elmer, <http://www.perkinelmer.com>). $I-V$ technique is measured with CuO-ZnO NRs fabricated flat AgE by using Keithley-Electrometer (<http://www.keithley.com>) from USA.

2.2. Synthesis and growth mechanism of CuO-ZnO NRs

CuO-ZnO NRs have been synthesized by adding uni-molar concentration of copper chloride and zinc chloride precursor into chemical reactors for 6 h. Copper chloride and zinc chloride were slowly dissolved separately into the de-ionized water to make

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