



Molybdenum disulfide nanosheets supported Au-Pd bimetallic nanoparticles for non-enzymatic electrochemical sensing of hydrogen peroxide and glucose



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ABSTRACT

The molybdenum disulfide (MoS₂) nanosheets supported Au-Pd bimetallic nanoparticles were synthesized using a facile thermal co-reduction method. The Au-Pd/MoS₂ nanocomposites showed good electrocatalytic activities toward the reduction of hydrogen peroxide (H₂O₂) in neutral solution and the oxidation of glucose in alkaline solution, attributed to the synergistic effects of the Au-Pd bimetallic nanoparticles and MoS₂ nanosheets. The fabricated non-enzymatic electrochemical sensors exhibited the linear responses of H₂O₂ and glucose in the concentration ranges of 0.8 μM–10 mM and 0.5–20 mM with the detection limits of 0.16 μM and 0.40 mM (S/N = 3), respectively, together with good stability and reproducibility. The Au-Pd/MoS₂ nanocomposites provide new potential materials for the development of other non-enzymatic sensors.

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1. Introduction

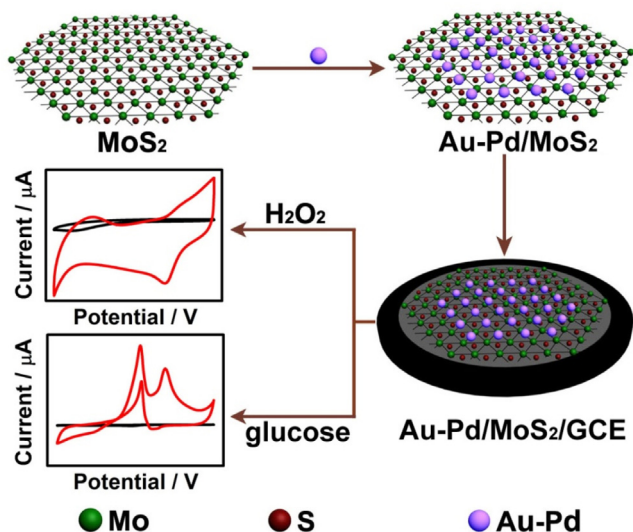
Hydrogen peroxide (H₂O₂) is an important intermediate in biological and environmental systems and has been widely used in various fields, such as pharmacy [1,2], environmental protection [3], and fuel cells [4]. H₂O₂ is also a major reactive oxygen species (ROS) [5] in various diseases, such as cancers, Parkinson's disease, and Alzheimer's disease [6,7]. Therefore, it is very essential to detect H₂O₂ rapidly and accurately. Glucose exists in human blood and is an important biological indicator associated with kidney dysfunction and failure. The fasting blood glucose concentrations are 3.9–6.1 mM for healthy people [8] and increase for diabetes, so accurate monitoring of glucose concentrations is of great importance to the treatment or control of diabetes. Although many techniques have been developed for the detections of H₂O₂ and glucose, the electrochemical technique is still deemed as the most popular analytical method, because of high sensitivity, low cost, and simple operation [9,10]. Most studies on the detections of the two species involve the use of enzymes [11,12], which catalyze the reduction of H₂O₂ and the oxidation of glucose. These enzymatic sensors have excellent sensitivity and selectivity, however,

the major problems of the enzymatic sensors are high cost and poor storage stability, due to the intrinsic thermal and chemical instability of these enzymes. To resolve these problems, many non-enzymatic sensors have been explored to improve the stability of H₂O₂ and glucose detections [13–18]. Therefore, it would be advantageous to prepare simple, rapid, and low-cost non-enzymatic sensors for the detections of H₂O₂ and glucose.

Recently, molybdenum disulfide (MoS₂), which is one of the most representative lamellar-structured transition-metal dichalcogenides [19], has received much attention in many fields, such as electrochemical sensors [20–22], catalysis [23], oxygen reduction reactions [24], hydrogen evolution [25,26], lithium-ion batteries [27], organic solar cells [28], and proton conducting membranes [29]. MoS₂ possesses typical lamellar structures, which are composed of three atomic layers with one layer of Mo atoms sandwiched between two layers of S atoms, and such three-sublayer stacking held together by weak van der Waals interactions [30]. A variety of strategies have been developed to allow bulk MoS₂ crystals to be exfoliated into mono- or a few layers of MoS₂ nanosheets by the intercalation of different types of ions or molecules [31]. The resulting MoS₂ nanosheets exhibit better properties than other two-dimensional (2D) analogues, such as semiconducting [32], photoluminescence [33,34], SERS-active substrates [35], and energy harvesting [36].

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Scheme 1. Illustration of preparation of Au-Pd/MoS₂ nanocomposites and their assembly on a glassy carbon electrode for non-enzymatic electrochemical determinations of H₂O₂ and glucose.

Herein, the unique physical and chemical properties of MoS₂ nanosheets and the high electrocatalytic activity of Au-Pd bimetallic nanoparticles motivated us to synthesize MoS₂ nanosheets supported Au-Pd bimetallic nanoparticles (Au-Pd/MoS₂) for fabrication of non-enzymatic sensors of H₂O₂ and glucose. The Au-Pd/MoS₂ nanocomposites were synthesized by a facile thermal co-reduction method and then used for the non-enzymatic electrochemical sensors of H₂O₂ and glucose (Scheme 1). The proposed non-enzymatic sensors showed wide concentration linear range, low detection limit, and good stability for the determinations of H₂O₂ and glucose.

2. Experimental section

2.1. Apparatuses and reagents

Morphologies of nanomaterials were acquired on a JEM-2100 transmission electron microscope (TEM) and an S-4800 scanning electron microscope (SEM), and surface elemental compositions were characterized by an energy-dispersive X-ray (EDX) spectroscopy. Raman spectra were collected on a Renishaw InVia Raman spectrometer equipped with a CCD detector, and a 532 nm laser was used for the excitation line. X-ray diffraction (XRD) patterns were measured on a PANalytical diffractometer (Philips, Netherlands) with a Cu-K α radiation ($\lambda = 0.15418$ nm). UV-vis spectra were recorded on a UV-3600 spectrophotometer (Shimadzu, Japan). All the electrochemical experiments were carried out on a CHI 660C electrochemical workstation (Shanghai Chenhua Equipment, China). A conventional three-electrode system was used with a modified glassy carbon electrode (GCE, 3 mm in diameter) as the working electrode, a saturated calomel electrode (SCE) as the reference electrode, and a platinum wire electrode as the counter electrode. Prior to each electrochemical measurement, supporting electrolytes were deoxygenated by bubbling nitrogen stream for at least 20 min to maintain the experiments under nitrogen atmosphere. All of the experiments were carried out at room temperature.

MoS₂ powder was purchased from Sigma-Aldrich, and ascorbic acid (AA) and uric acid (UA) were from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China). Palladium acetate (Pd(OAc)₂) was obtained from Shanghai Kefeng Industry & Commerce Co.,

Ltd. (Shanghai, China). Chloroauric acid (HAuCl₄·4H₂O), trisodium citrate, and inorganic salts (KCl, NaNO₃, Na₂SO₄, and Na₂CO₃) were purchased from Nanjing Chemical Reagent Co., Ltd. (Nanjing, China), and 4-acetamidophenol (AP) was from Aladdin. Phosphate buffered saline (PBS, 0.01 M, pH 7.4) and aqueous NaOH solution (0.1 M) were used as supporting electrolytes for the detections of H₂O₂ and glucose, respectively. All other reagents were of analytical grade and used without further purification. The double-distilled water with a resistivity of 18.2 M Ω cm was used for all of the experiments.

2.2. Preparation of Au-Pd/MoS₂ nanocomposites

MoS₂ powder (30 mg) was added to a 25-mL flask followed by addition of 10 mL of ethanol/water (1:1, v/v), and the mixture was ultrasonicated with a sonicator (180 W, 40 kHz) for 8 h followed by centrifugation at 3000 rpm for 20 min three times to remove aggregates [37]. The as-synthesized MoS₂ nanosheets were suspended in double-distilled water at the concentration of 1.6 mg mL⁻¹. The supernatant was collected for UV-vis spectral measurement.

The Au-Pd/MoS₂ nanocomposites were synthesized according to the method for synthesis of reduced graphene oxide supported Au-Pd bimetallic nanoparticles [38], but with different amounts of relevant reagents. Trisodium citrate (0.135 g) was added to 22.5 mL of aqueous dispersion of MoS₂ nanosheets (0.5 mg mL⁻¹). The mixture solution was ultrasonicated at room temperature for approximately 30 min until a homogeneous solution was obtained. Next, 4.3 mg of Pd(OAc)₂, 0.25 mL of aqueous HAuCl₄ solution (24 mM), and 1.15 mL aqueous AA solution (0.1 M) were sequentially added to the above mixture solution. The mixture was heated under magnetic stirring at 100 °C for 2.5 h, and then the color turned black. After cooling down to room temperature, the Au-Pd/MoS₂ nanocomposites were collected by centrifugation and then repeatedly washed with double-distilled water and ethanol several times.

2.3. Preparation of Au-Pd/MoS₂/GCE sensors

A GCE was sequentially polished to a mirror-like shine with 1.0, 0.3, and 0.05 μ m alumina slurries and then rinsed thoroughly with double-distilled water after each step. Then, the electrode was sequentially ultrasonicated in ethanol and double-distilled water followed by drying with nitrogen stream. Finally, 7 μ L of the aqueous dispersion of Au-Pd/MoS₂ composites was dropped onto the surface of the treated GCE by a pipette followed by drying in a vacuum desiccator at least 2 h (denoted as Au-Pd/MoS₂/GCE). Other modified electrodes were prepared with aqueous dispersions of corresponding nanomaterials.

2.4. Determinations of H₂O₂ and glucose

Differential pulse voltammetry (DPV) was used to determine H₂O₂ in the potential range from -0.35 to 0.2 V versus SCE at a scan rate of 0.1 V s⁻¹ in PBS solution (pH 7.4). Chronoamperometry was used to determine glucose at the potential of -0.1 V in aqueous NaOH solution (0.1 M). Each sample was repeatedly measured for three times. The error bars represented the standard deviation of the triplicate measurements.

3. Results and discussion

3.1. Characterizations of Au-Pd/MoS₂ nanocomposites

The as-synthesized MoS₂ nanosheets looked like silk veil and were entangled with each other (Fig. 1A). The high-resolution TEM (HRTEM) image (inset of Fig. 1A) showed that the lattice spacings of 0.279 nm between neighboring fringes were the parallel

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