



A facile fabrication of platinum nanoparticle-modified graphite pencil electrode for highly sensitive detection of hydrogen peroxide



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ABSTRACT

A novel platinum nanoparticle-modified graphite pencil electrode (PtNP-GPE) for a non-enzymatic determination of H₂O₂ is proposed. The PtNP-GPE is prepared by heating a GPE in an aqueous solution of ammonium tetrachloroplatinate(II) and ascorbic acid at 75 °C for 15 min. Both unmodified and PtNP-GPE are characterized using field-emission-SEM and cyclic voltammetry. The PtNP-GPE showed better electrocatalytic properties toward the electrochemical redox reaction of H₂O₂ than that of the unmodified GPE. The PtNP-GPE amperometric response is tested at four different potentials (+0.7, +0.5, −0.2 and −0.3 V vs. Ag/AgCl reference electrode). Taking into consideration both the intensity of the amperometric response and detector stability, the applied potential of +0.5 V is the best among the tested potentials. The PtNP-GPE amperometric response of H₂O₂ at +0.5 V is linear from 10 to 110 μM (R² = 0.999) with a detection limit of 3.6 μM.

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1. Introduction

Hydrogen peroxide (H₂O₂) is commonly used in wide areas such as textile, cleaning products, food industry, organic compounds, and effluent treatments. As a result, H₂O₂ is haphazardly distributed in the environment especially in water and industrial products. The toxicity of H₂O₂ to living organisms is notable [1]. On the other hand, it is a signal generating molecule in many biosensors, e.g. enzyme-based glucose and lactate sensors [2–4]. In enzyme-based glucose sensors, glucose oxidase produces H₂O₂ from glucose and oxygen [2,3]. The generated H₂O₂ was quantified to determine the glucose concentration indirectly [2,3]. Therefore, simple, cheap, quick, accurate and reliable methods for the sensitive detection of hydrogen peroxide are of interest to many fields; such as food and food additives, cosmetics, pharmaceutical's products, environmental analysis and biosensor fabrication. Up to date, various methods were developed to detect H₂O₂ based on titrimetric, spectrophotometric, fluorescence, chemiluminescence and electroanalytical methods [5–9]. Among those; electroanalytical methods have attracted much attention due to its high sensitivity, portability, and short response time and low cost.

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For the last two decades, nanomaterial-modified electrodes are widely applied as transducers in various fields like sensors due to their better physical, chemical, electrochemical properties and higher surface area compared to their counter bulk materials [10–13]. Particularly, many research groups applied platinum nanoparticle-modified electrodes for the electrochemical detection of H₂O₂ as the PtNP-modified electrodes have shown impressively electrocatalytic properties toward H₂O₂ [3,14–17]. However, using of PtNP-modified electrode for often detection of H₂O₂ is limited due to the high cost. The cost of the PtNP-modified electrode depends on the used substrate as well as on the PtNP attachment/preparation method. Among substrates, the graphite pencil electrode (GPE) is the cheapest and most available one. Nevertheless, it shows poorly electrocatalytic properties towards many electroactive molecules [18,19]. To improve the electrocatalytic properties of the GPEs, various approaches for either pretreatment [20,21] or modifications [22–27] were reported. Since a high electrocatalytic property is required to fabricate sensitive electrochemical sensors, modification of GPE with PtNP is logic to make a high electrocatalytic electrode for the sensitive detection of hydrogen peroxide. For the modification of GPE with metal or metal oxide NP, chemical vapor deposition from metal precursor and electrochemical deposition from polymer-attached NPs were reported [24,28–30]. Both chemical vapor deposition and electrochemical methods are extremely fast processes, thus generate irreproducible surfaces where a reproducible surface is essential to make an

efficient sensor. Moreover, such methods require sophisticated instruments and quite skilled persons that increase the overall sensor fabrication cost. As a result, development of a simple chemical method for the preparation of PtNP-modified GPE is quite meaningful.

Recently, Compton et al. developed facile methods for the preparation of Pt, Ag, PdNP-modified glassy carbon (GC) microsphere in a solution phase using correspondence metal precursors and a reductant (ascorbic acid (AA) or hydrazine) in the absence or presence of stabilizers at elevated temperatures [31,32].

To the best of our knowledge, there are no reports for a single-step chemical preparation of PtNP-modified GPE using a metal precursor, reductant and GPE. The proposed PtNP-GPE is a disposable, sensitive and selective hydrogen peroxide detector combining the advantages of PtNP and GPE. The morphological characterization of the fabricated PtNP-GPE is shown in the following sections along with its electrochemical characterization, to explore its electrocatalytic properties toward redox reaction of H_2O_2 .

2. Experimental

2.1. Instrumentation

UV experiments were performed using Ocean Optics USB4000-UV-VIS Spectrophotometer (Dunedin, FL, USA). A Jedo mechanical pencil (Korea) was used as a holder for both bare and PtNP-modified graphite pencil leads. Electrical contact with the lead was achieved by soldering copper wire to the metallic part that holds the lead in place inside the pencil. The pencil was fixed vertically with 15 mm of the pencil lead extruded outside, and 10 mm of the lead immersed in the solution. Such length corresponds to a geometric electrode area of 15.90 mm^2 . Details of the pencil electrode were described earlier [33]. CHI 660C (CH Instruments Inc., Austin, TX, USA) was used for the entire electrochemical work. The electrochemical cell contained bare- or PtNPs-modified GPE as a working electrode, a Pt wire counter electrode and Ag/AgCl (Sat. KCl) reference electrode. Field emission scanning electron microscopic (FE-SEM) images are recorded using TESCAN LYRA 3 (Brno, Czech Republic) at the Center of Research Excellence in Nanotechnology, King Fahd University of Petroleum and Minerals, Kingdom of Saudi Arabia.

2.2. Chemicals and reagents

Ammonium tetrachloroplatinate(II) ($(NH_4)_2PtCl_4$), L-ascorbic acid (AA), uric acid (UA), 4-acetamidophenol (AAP), dopamine (DA), hydrogen peroxide (H_2O_2) and sodium hydroxide (NaOH) were purchased from Sigma-Aldrich (St. Louis, MO, USA). Disodium hydrogen phosphate and sodium dihydrogen phosphate were supplied by Fisher Scientific Company (Pittsburgh, PA, USA). Hi-polymer graphite pencil HB black leads were obtained from Pentel Co., Ltd. (Japan). All leads had a total length of 60 mm and a diameter of 0.5 mm, and were used as received. All solutions were prepared with deionized water of resistivity of $18.6 \text{ M}\Omega \text{ cm}^{-1}$, which was obtained directly from PURELAB[®] Ultra Laboratory Water Purification System (Siemens, Washington, DC, USA).

2.3. Preparation of PtNP-modified graphite pencil electrode

Equal volumes of 1.5 mL aqueous solution of 1.1 mM AA and 1.0 mM $(NH_4)_2PtCl_4$ were mixed using a pipette at room temperature (RT) in 3.0 mL test tube. Afterward, a bare GPE was immersed into a 3.0 mL test tube containing the freshly prepared solution of $(NH_4)_2PtCl_4$ and AA. To fabricate the PtNPs-modified GPE, that test tube was placed into a water bath preheated to 75°C and kept for

15 min. The PtNPs-modified GPE was removed and washed by gentle dipping two times into deionized water, then dried at 60°C for 5 min prior to use.

3. Results and discussion

3.1. Reductive capacity of AA to form PtNP from $[PtCl_4]^{2-}$

It is reported that AA or salt of AA is a good reductant to form AuNP quickly at room temperature (RT) from gold precursor [34]. Besides, Compton group prepared PtNP on GC microsphere by stirring the mixture of GC microsphere, precursor of Pt and AA at 70°C for 2 h with no explanation for the necessity of heat treatment [25]. In the present work, UV experiments are performed initially to understand the necessity of heat treatment to form PtNP using AA as a reductant. Fig. 1(i) and (ii) shows the UV spectra of 0.5 mM $(NH_4)_2PtCl_4$ (aq.) and 0.5 mM AA (aq.), respectively.

The AA shows almost no absorbance within the tested wavelength range (320–700 nm), where $(NH_4)_2PtCl_4$ shows absorbance between 360 to 545 nm with λ_{max} 387 nm. Fig. 1(iii) and (iv) are the UV spectra of the aqueous solution of 0.5 mM $(NH_4)_2PtCl_4$ and 0.5 mM AA. Fig. 1(iii) is recorded spectrum right after making the solution, whereas spectrum (iv) is for the same prepared solution after 15 min at RT. There are almost no differences among spectra (i), (iii) and (iv) of Fig. 1, i.e. AA does not reduce $PtCl_4^{2-}$ at RT within 15 min period. The freshly prepared aqueous solution mixture of 0.5 mM $(NH_4)_2PtCl_4$ and 0.55 mM AA is colorless, and with heating at 75°C started to turn to light black (grey color). The UV spectrum of the 15 min heated reaction mass (Fig. 1(v)) shows a high absorbance without any peaks. The UV spectrum (Fig. 1(v)) is characteristics for the aqueous solution of PtNP [35,36] i.e. AA can easily reduce the $PtCl_4^{2-}$ to form PtNP at elevated temperatures.

3.2. Preparation and characterization of the PtNP-modified GPE

In the previous section, we concluded that AA could not reduce $PtCl_4^{2-}$ at RT within 15 min in the absence of the GPE. However, that possibility is there in the presence of GPE as it composed of graphite and clay. The catalytic growth of PtNP on any of the GPE components at RT from the aqueous solution of $PtCl_4^{2-}$ and AA is a concern. To check the catalytic growth of PtNP, initially GPE is immersed in the aqueous solution of 0.5 mM $(NH_4)_2PtCl_4$ and 0.55 mM AA for 15 min at RT. Afterward, the RT treated electrode was subjected to record FE-SEM images (Fig. 2b) and cyclic voltammogram (CV)

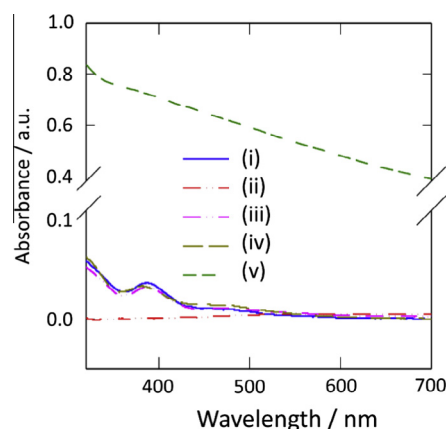


Fig. 1. UV-visible spectra of an aqueous solution of (i) 0.5 mM $(NH_4)_2PtCl_4$, (ii) 0.55 mM AA, (iii) 0.5 mM $(NH_4)_2PtCl_4$ and 0.55 mM AA (just after preparation), (iv) 0.5 mM $(NH_4)_2PtCl_4$ and 0.55 mM AA after 15 min at room temperature for (v) 0.5 mM $(NH_4)_2PtCl_4$ and 0.55 mM AA after heating at 75°C for 15 min.

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