



A non-enzymatic hydrogen peroxide sensor based on platinum nanoparticle–polyaniline nanocomposites hosted in mesoporous silica film



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ABSTRACT

We demonstrated a facile method for supporting platinum nanoparticles (PtNPs) on polyaniline (PANI)/mesoporous silica film (MSF). The MSF with mesochannels perpendicular to the underlying electrode surface can confine the electrodeposition of PANI and improve the mechanical strength of PANI. The secondary amines and tertiary imines on the PANI backbone can readily complex with PtCl₆²⁻ ions, which can be further reduced to generate Pt nanoparticles (NPs). The obtained PtNPs@PANI/MSF hybrid material was characterized by transmission electron microscopy and energy dispersive X-ray spectroscopic analysis. It also exhibited a good electrocatalytic activity toward oxidation of H₂O₂ and can be used to detect H₂O₂ with a high sensitivity (50 μA mM⁻¹) in a wide concentration range (1.0–2000 μM).

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1. Introduction

Metal nanoparticles (NPs) have continued to receive considerable interest due to the distinctly different physicochemical properties compared to their bulk counterparts, which led to numerous potential applications in optical, electronic, catalysis and sensing [1–2]. In particular, a lot of research works have concentrated on platinum (Pt) and Pt-alloys because of their high activities [3–5]. However, most of the metal NP based catalysts may undergo aggregation or suffer from poisoning under the reaction conditions, resulting in the deactivation and loss of catalytic activity [6–7]. Addition of organic ligands or preparation of supported metal NPs can effectively solve this problem [8–9]. For example, metal NPs can be dispersed on various porous materials, including metal oxides, carbon and polymers [10–13]. Moreover, a suitable supporting material may have a significant effect on the catalytic activity owing to the interactions and surface reactivity with supported NP catalysts [14]. Among them, conducting polymer supported metal NPs have attracted many research efforts, due to the synergetic properties pertaining of the two components [15–18]. So far, there are many reports on loading PtNPs on conducting polymers for applications in electrochemical sensors

[2,24], dye-sensitized solar cell [25], regioselective hydrosilylation reactions [26] and so on.

Herein we report a facile procedure to support PtNPs on polyaniline (PANI) spatially confined in channels of mesoporous silica film (MSF). PANI is one of the most studied conducting polymer hosts due to its facile synthesis, controllable electronic conductivity, relatively high chemical stability and potential applications in many areas [19–23]. Confining PANI in the pores of MSF can improve the poor mechanical strength of conducting polymers [27–29] and increase the surface-to-volume ratio [30]. After being dedoped in an alkaline solution, the secondary amines and tertiary imines on the PANI backbone can readily complex with PtCl₆²⁻ ions. Upon further chemical reduction with NaBH₄, PtNPs were produced and loaded onto the PANI–MSF. Thus obtained hybrid material, designated PtNPs@PANI–MSF, exhibited a good electrocatalytic activity and sensing capability toward hydrogen peroxide (H₂O₂).

2. Materials and methods

2.1. Chemicals and materials

All chemicals used were analytical grade or higher. Perchloric acid (HClO₄), potassium perchlorate (K₂PtCl₆), concentrated ammonia aqueous solution (25 wt%), potassium hydrogen phthalate (KHP) and aniline were ordered from Aladdin. Hydrochloric

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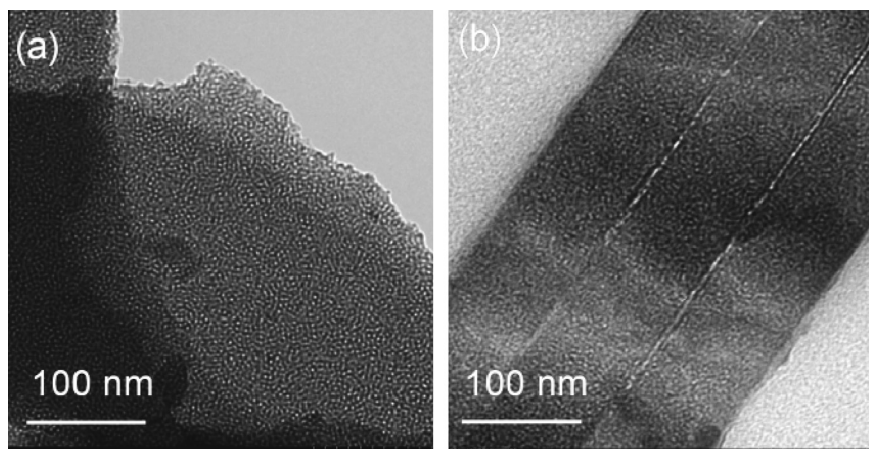


Fig. 1. TEM images of MSF with top (a) and cross-section (b) view.

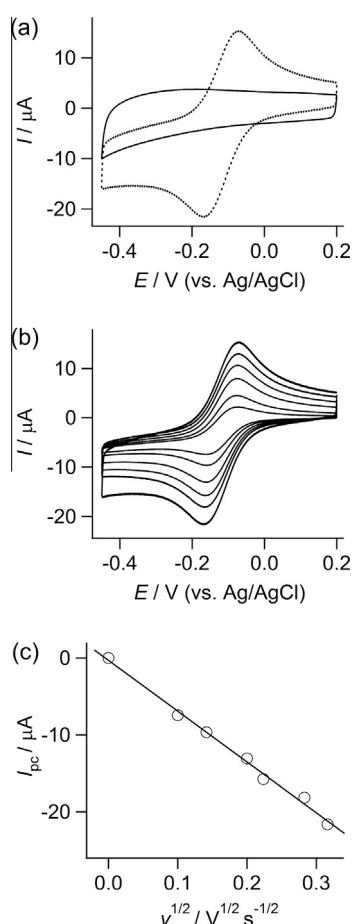


Fig. 2. (a) Cyclic voltammograms of a MSF modified ITO electrode before (solid line) and after (dot line) surfactant removal in 0.05 M KHP (pH = 4.0) containing 50 μM $\text{Ru}(\text{NH}_3)_6\text{Cl}_3$. The scan rate was 0.1 V s^{-1} . (b) Cyclic voltammograms of a MSF modified ITO electrode at various scan rates: 0.01, 0.02, 0.04, 0.06, 0.08 and 0.1 V s^{-1} from inner to outer. (c) The dependence of cathodic peak current on the square root of scan rate.

acid (HCl) was obtained from Hangzhou Chemicals. Tetraethoxysilane (TEOS), cetyltrimethylammonium bromide (CTAB) and hexammineruthenium (III) chloride ($\text{Ru}(\text{NH}_3)_6\text{Cl}_3$, 98%) were purchased from Sigma-Aldrich. Sodium borohydride (NaBH_4) was bought from Alfa Aesar. Aniline was distilled at reduced pressure prior to use. Anhydrous ethanol, acetone and sodium hydroxide

(NaOH) were supplied by Sinopharm. Indium tin oxide (ITO) coated glass (thickness of 100 nm, resistance of $<15 \Omega/\text{square}$) was ordered from Zhuhai Kaivo Electronic Components. Prior to use, the ITO glass was treated with 1 M NaOH solution overnight to remove the organic residues, and then sonicated in acetone, ethanol and deionized water sequentially for 10 min. Finally, cleaned ITO glasses were dried with a nitrogen stream. Deionized water ($18.2 \text{ M}\Omega \text{ cm}$) was used in all experiments.

2.2. Preparation of MSF and PtNPs@PANI-MSF

MSF was synthesized on ITO electrode with stöber-solution method as previously reported [31]. Typically, ITO electrodes were immersed in solution containing 0.16 g cetyltrimethylammonium bromide (CTAB), 70 mL water, 30 mL ethanol, 10 μL ammonia aqueous solution and 80 μL tetraethoxysilane (TEOS). After 24 h, MSF coated ITO electrodes were taken out, rinsed with water and dried at 100°C overnight. Prior to use, the surfactants CTAB were removed in an ethanol solution containing 0.1 M HCl under stirring for 5 min. The PANI-MSF was prepared by electrodeposition of aniline in the channels of MSF as demonstrated in our previous work [32]. After being dedoped in 0.1 M $\text{NH}_3\cdot\text{H}_2\text{O}$, the PANI-MSF was immersed in 5 mM K_2PtCl_6 aqueous solutions for 24 h to complex with PtCl_6^{2-} . Eventually, the chemical reduction of PtCl_6^{2-} by 0.1 M NaBH_4 led to the deposition of PtNPs in PANI-MSF, and thus obtained hybrid material is designated PtNPs@PANI-MSF. The ITO electrodes modified by this hybrid material were dried at 60°C under vacuum for further study.

2.3. Instrumentation

All electrochemical experiments were carried out on a CHI 660D electrochemical workstation (Chenhua Instruments, Shanghai). A traditional three-electrode system was utilized, with the MSF coated ITO glass as the working electrode, a platinum wire and Ag/AgCl (in saturated KCl) as the counter and reference electrodes, respectively. Chronoamperometry was performed in PBS solution (0.1 M, pH 7.4). All the measurements were implemented at room temperature ($20 \pm 2^\circ\text{C}$).

Transmission electron microscopy (TEM) images were obtained on a HT7700 transmission electron microscope (Hitachi, Japan) at an accelerating voltage of 120 kV. Energy dispersive X-ray spectroscopic (EDX) analysis was performed on a field emission transmission electron microscope (FE-TEM) equipped with an energy dispersive X-ray spectrometer (EDX) (JEM-2100F, Japan) operated at 200 kV. The TEM and EDX samples were prepared by

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