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One-pot synthesis of poly(3,4-ethylenedioxythiophene)-Pt nanoparticle composite and its application to electrochemical H₂O₂ sensor

Li-Chi Chang¹, Huan-Nung Wu¹, Chia-Yu Lin¹, Yi-Hsuan Lai¹, Chih-Wei Hu¹ and Kuo-Chuan Ho^{1,2*}**Abstract**

Poly(3,4-ethylenedioxythiophene)-Pt nanoparticle composite was synthesized in one-pot fashion using a photo-assisted chemical method, and its electrocatalytic properties toward hydrogen peroxide (H₂O₂) was investigated. Under UV irradiation, the rates of the oxidative polymerization of EDOT monomer along with the reduction of Pt⁴⁺ ions were accelerated. In addition, the morphology of PtNPs was also greatly influenced by the UV irradiation; the size of PtNPs was reduced under UV irradiation, which can be attributed to the faster nucleation rate. The immobilized PtNPs showed excellent electrocatalytic activities towards the electroreduction of hydrogen peroxide. The resultant amperometric sensor showed enhanced sensitivity for the detection of H₂O₂ as compared to that without PtNPs, i.e., only with a layer of PEDOT. Amperometric determination of H₂O₂ at -0.55 V gave a limit of detection of 1.6 μM (S / N = 3) and a sensitivity of 19.29 mA cm⁻² M⁻¹ up to 6 mM, with a response time (steady state, t₉₅) of 30 to 40 s. Energy dispersive X-ray analysis, transmission electron microscopic image, cyclic voltammetry (CV), and scanning electron microscopic images were utilized to characterize the modified electrode. Sensing properties of the modified electrode were studied both by CV and amperometric analysis.

Keywords: H₂O₂ electrochemical sensors, Photochemical assisted deposition, Poly (3,4-ethylenedioxythiophene), Pt nanoparticles

Background

Research on the quantitative detection of hydrogen peroxide (H₂O₂) received considerable attention because H₂O₂ is widely used as an oxidizing agent in chemical and food industries [1]. It is an essential mediator in food, pharmaceutical, clinical, and environmental analysis. In addition, H₂O₂ is produced during some chemical and enzymatic processes [2,3]; its detection can be used as an indicator for the progress of such processes. Among the developed methodologies [4-7], electrochemical technique is an appropriate alternative or a complementary choice since it has been proved to be an inexpensive and effective way for quantitative determination owing to its intrinsic

sensitivity, fast analysis, high selectivity and simplicity. H₂O₂ can be detected anodically at a platinum electrode at around +0.7 V vs. SCE; it can also be detected cathodically at a copper electrode at -0.25 V vs. SCE [1].

Many electrode materials, including Pt [8,9], Ag [10], Cu [11], and Prussian blue [12], have been explored as electrocatalysts for the detection of H₂O₂. Among these materials, Pt shows excellent electrocatalytic activity towards H₂O₂. Recent studies [13-15] show that the improvement in electrocatalytic activity of Pt, in terms of overpotential and sensitivity, can be achieved by the use of the nanosized or nanostructured Pt as compared with its bulk counterpart, owing to its extraordinary surface properties and larger specific surface area. In addition, it has also been reported that the size and shape of Pt play an important role in determining the electrocatalytic activity for H₂O₂ [16].

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The use of conducting polymers as electrode materials in the field of electrocatalysis [17-19] has been a hot research topic not only because they, themselves, exhibit electrocatalytic properties toward many important analytes but also act as effective electrocatalyst support. Regarding the latter, conducting polymers not only can provide sufficient accessible surface area, low resistance, and high stability but also induce uniform distribution of metal nanoparticles and facile electron transfer between electrocatalysts and electrode. Poly(3,4-ethylenedioxythiophene) (PEDOT) has become one of the most intensively studied conducting polymers due to its excellent conductivity, chemical stability, and electrocatalytic properties. In addition to its potential application for the detection of important analytes, such as dopamine [20], nitrite [21], and ascorbic acid [22], the pristine PEDOT and its composite with Pt have also been explored in the fields of fuel cells [23-25], photovoltaics [26-28], and super-capacitors [29,30].

This study reports the preparation of a modified electrode and its application as a sensor for the amperometric detection of H_2O_2 based on a screen-printed carbon (SPC)

electrode using a composite film of PEDOT and PtNPs designated as PEDOT-PtNPs/SPC electrode. Although there have been reports on the composite film of PtNPs with PEDOT for fuel cell applications [23-25], there is no report on the synthesis of the composite of PEDOT with PtNPs by photo-assisted chemical method and its application for sensing hydrogen peroxide. PEDOT-PtNPs/SPC electrode was prepared firstly by synthesizing PEDOT-PtNP composite in one-pot fashion using a photochemical polymerization method and, subsequently, depositing the composite onto the SPC electrode via the drop-coating method. Cyclic voltammetry (CV) technique was used to study the catalytic reduction of H_2O_2 on the PEDOT-PtNPs/SPC electrode. The potential use of the PEDOT-PtNPs/SPCE electrode for the amperometric detection of H_2O_2 was discussed.

Methods

Chemicals and instruments

3,4-Ethylenedioxythiophene (EDOT, 98%) and chloroplatinic acid hydrate (>99.5%) were purchased from

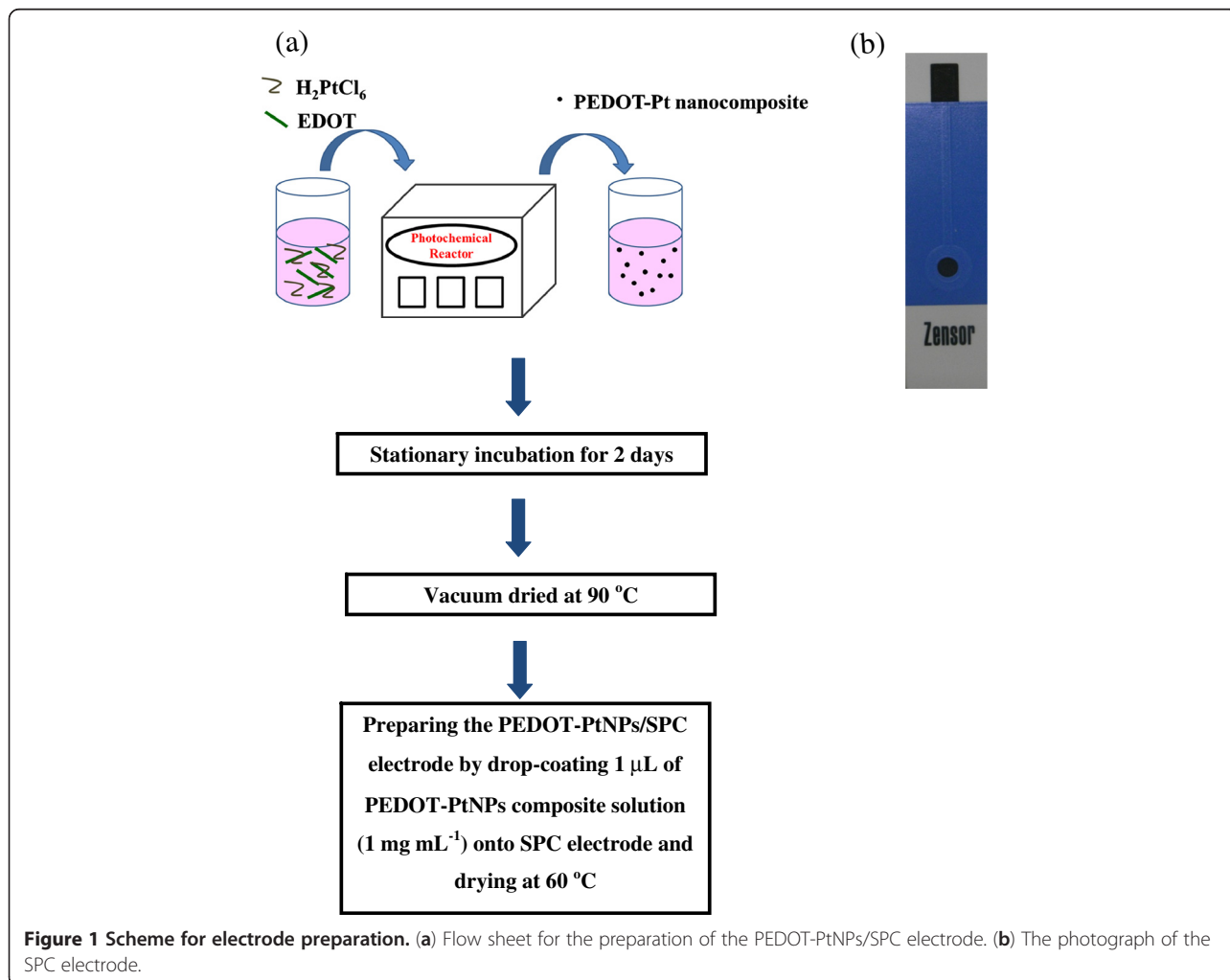
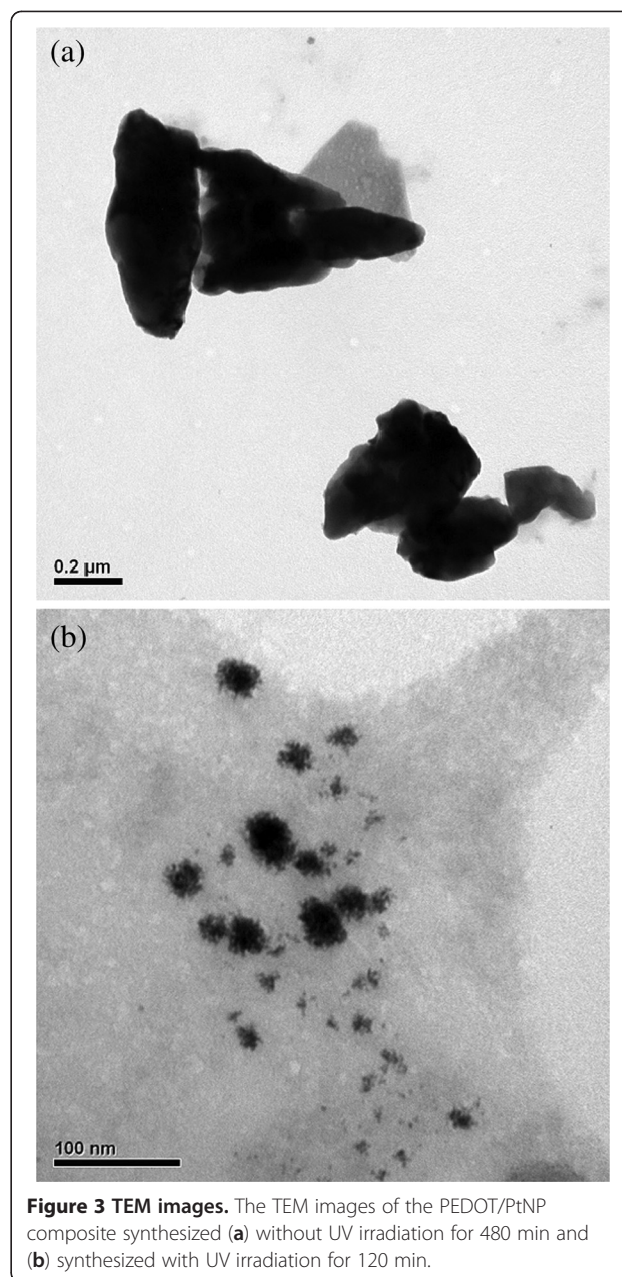
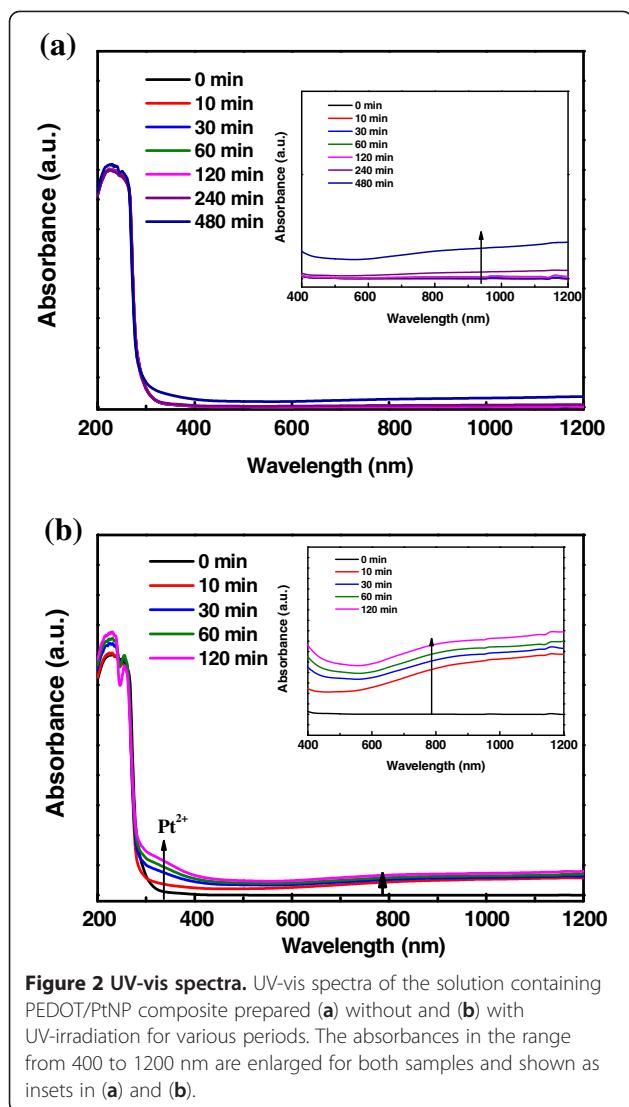


Figure 1 Scheme for electrode preparation. (a) Flow sheet for the preparation of the PEDOT-PtNPs/SPC electrode. (b) The photograph of the SPC electrode.

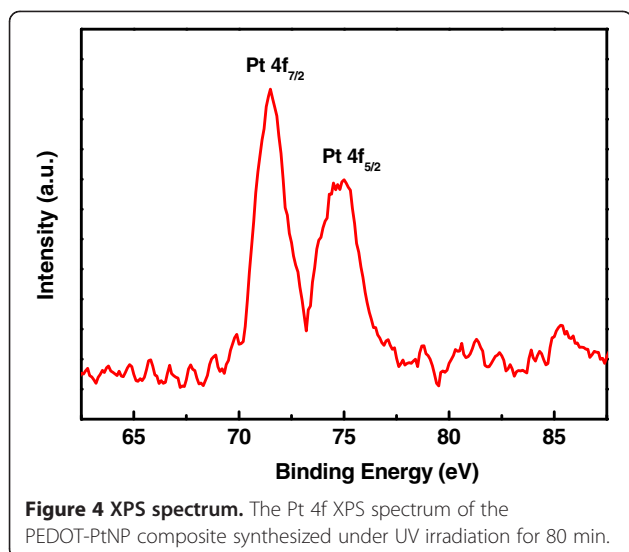


Sigma-Aldrich Corporation (St. Louis, MO, USA) and used as received. Dimethylsulfoxide (DMSO, 99.7%; Sigma-Aldrich) was dehydrated with molecular sieves (4 Å; Acros Organics, New Jersey, USA) before use. H₂O₂ sample solution (50 mM) was prepared before each experiment by direct dilution of H₂O₂ (35%; Sigma-Aldrich) in deionized water (DIW) and deaerated by purging it with nitrogen for 20 min. Other chemicals were of analytical grade and used without further purification. DIW was used throughout the work.

Electrochemical measurements were carried out using a CHI 440 electrochemical workstation (CH Instruments, Inc., USA) with a conventional three-electrode system; A SPC electrode with a geometric area of 0.071 cm² (Zensor R&D, Taiwan), Ag/AgCl/KCl saturated, and Pt foil are the working electrode, reference electrode, and counter electrode, respectively. All electrochemical experiments were performed at room temperature and all the potentials

are reported against the Ag/AgCl/KCl saturated reference electrode.

The nanoscale image of PEDOT-PtNP composite was obtained using scanning electron microscope (SEM, Nova NanoSEM 230, FEI Company, USA); elemental analysis was made using the same SEM with an additional provision of *x*-sight light element energy dispersive X-ray (EDX) spectrometer (6560 INCA, Oxford Instruments, UK). Transmission electron microscopy (TEM, H-7100, Hitachi Ltd., Japan) was also used to obtain the image of PEDOT-PtNP composite. The oxidation state of PtNPs was determined by X-ray photoelectron spectroscopy using an X-ray recorded on a PHI 5000 VersaProbe (ULVAC-

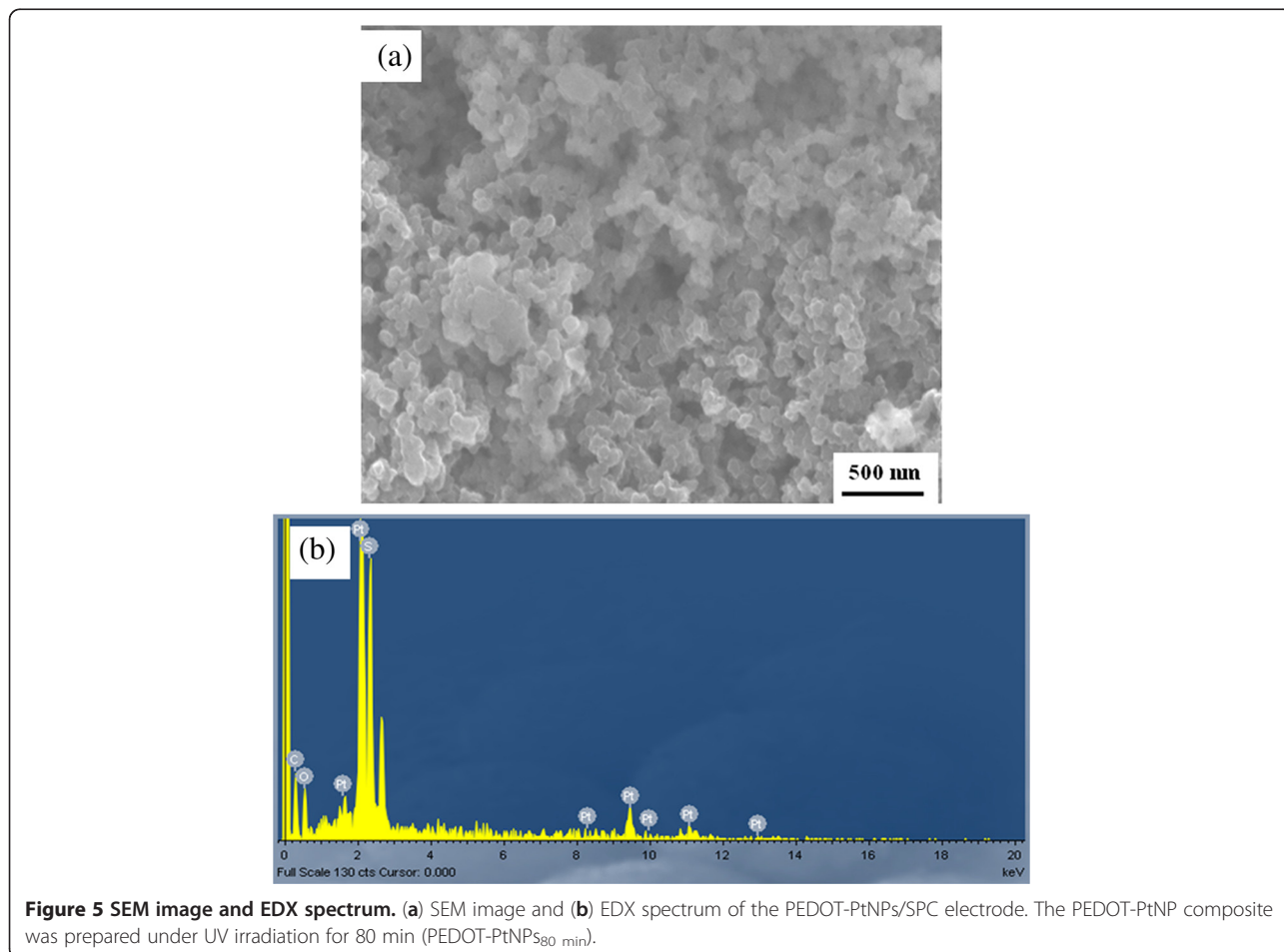


PHI, Inc., Chigasaki, Japan) system using a micro-focused (100 μm , 25 W) Al X-ray beam. A Wien-filtered C_{60}^+ ion source (IOG C60-10, Ionoptika Ltd., Chandler's Ford, UK) was operated at 10 nA and 10 kV. The angle between the

Ar^+ and C_{60}^+ ion beam was 33° . The ion-beam current was measured with the target current of a Au foil. The base pressure of the main chamber ($<1 \times 10^{-7}$ Pa) was maintained using turbomolecular and ion-getter pumps.

Preparation of the PEDOT-PtNPs/SPC electrode

The synthesis of PEDOT-PtNP composite was carried out by putting a glass bottle, containing 0.01 M of EDOT monomer and 0.001 M of H_2PtCl_6 aqueous solution, in a photochemical reactor (Panchum Scientific Corp., Taiwan) followed by irradiation with UV light (power density, 0.14 W cm^{-2} ; main wavelength, 365 nm) for a specific period of time under forced air convection and mild agitation. As the reaction proceeded, a black-colored suspension was obtained. After UV irradiation, the glass bottle containing black suspension was removed from the photochemical reactor and kept in the dark place at room temperature over 2 days for the precipitation of the composite; after which, the supernatant was removed, and the precipitate was subjected to vacuum dried at 90°C . After being dried, the precipitate was dispersed in DMSO solution at a concentration of 1.0 mg mL^{-1} . To prepare the PEDOT-PtNPs/SPC electrode, $1.0 \mu\text{L}$ of the dispersion was drop-coated onto the



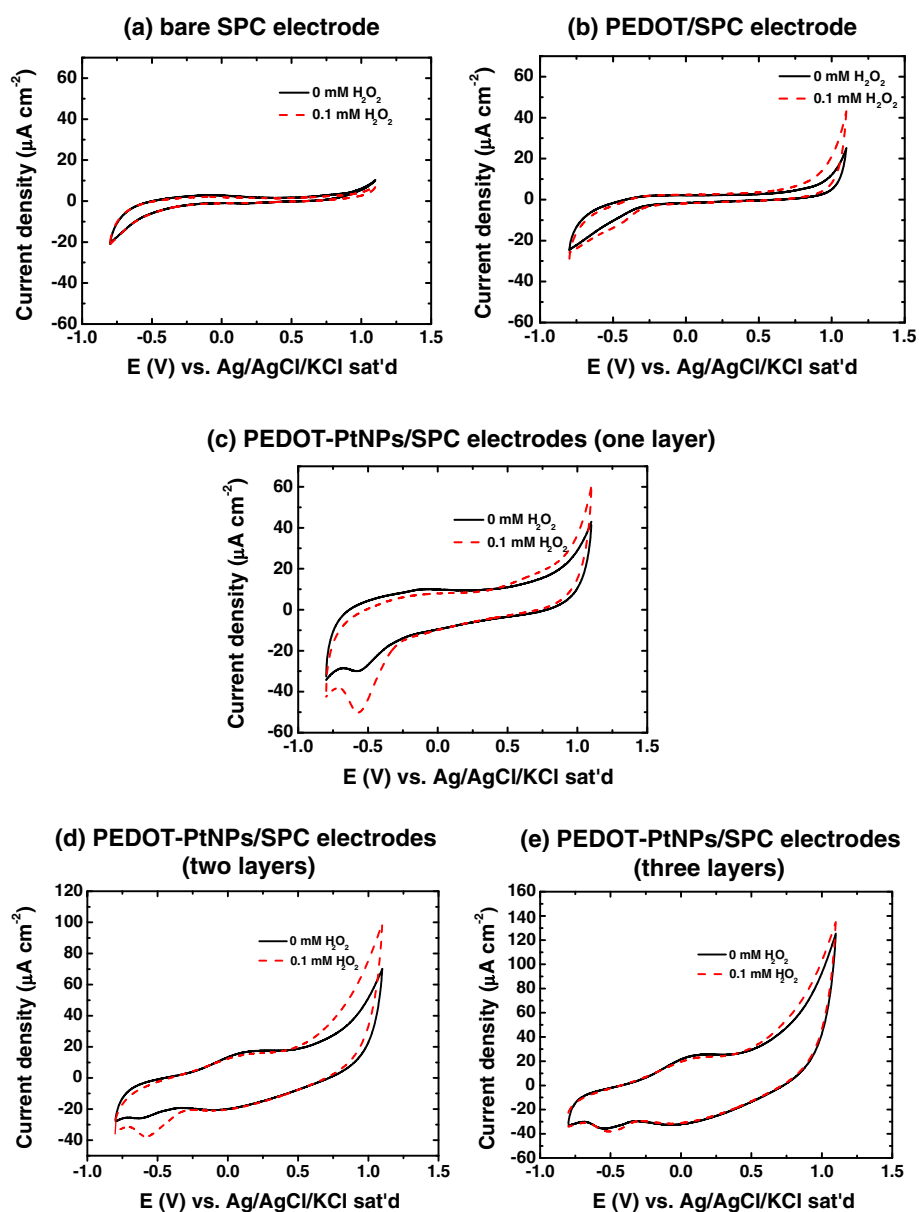


Figure 6 Cyclic voltammetry. Cyclic voltammograms of the (a) bare SPC, (b) PEDOT/SPC, (c) PEDOT-PtNPs/SPC electrodes (one layer), (d) PEDOT-PtNPs/SPC electrodes (two layers), and (e) PEDOT-PtNPs/SPC electrodes (three layers) in 0.1 M phosphate buffer solution (pH 7.4) with and without adding 0.1 mM H_2O_2 . Scan rate is 25 mV s^{-1} .

SPC electrode, and the coated SPC electrode was dried at 60°C . The scheme of the preparation of PEDOT-PtNPs/SPC electrode is showed in Figure 1.

Amperometric detection of H_2O_2

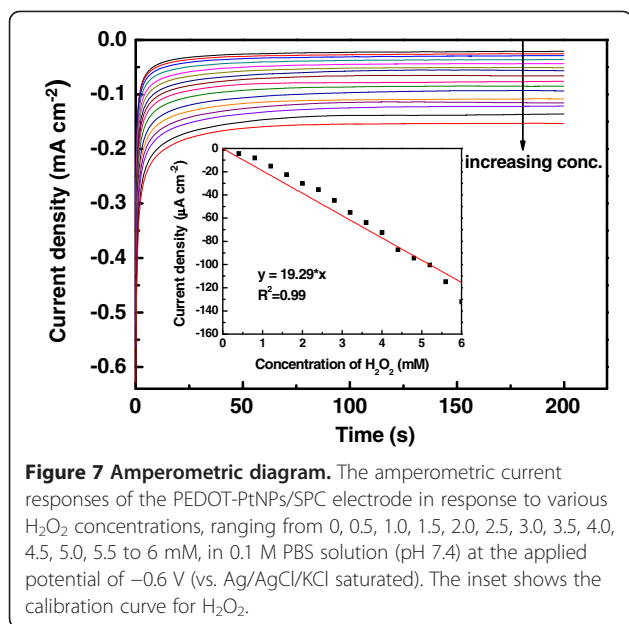
For the amperometric detection of H_2O_2 with amperometry at constant potential by using PEDOT-PtNPs/SPC electrode as the sensor, a suitable sensing potential in the limiting current plateau ranging between 0 and -0.7 V was determined by applying linear sweep voltammetry in a solution containing deaerated 0.1 M PBS (pH 7.4) and 0.5 mM

H_2O_2 (not shown). Considering the sensitivity and the steadiness of the PEDOT-PtNPs/SPC electrode, the sensing potential was chosen to be -0.6 V . Current densities in the concentration range of 0.4 to 6 mM were collected, and the pertaining calibration curve was constructed for the detection of H_2O_2 .

Results and discussion

Characterization

Figure 2 shows the UV-vis spectra of the PEDOT-PtNP composite prepared with and without UV irradiation for



various periods. The UV-vis spectra were recorded using PEDOT-PtNP composite dispersion prepared by dispersing a 0.1 mL of the reaction solution in the DMSO solvent with the help of sonication. It can be found that the absorbances at wavelengths between 310 and 380 nm, and at wavelengths higher than 700 nm increased monotonically during the time course of the reaction. The increase in the absorbance at wavelengths higher than 700 nm was assigned to the polymerization of EDOT [31]. Therefore, the polymerization rate of EDOT film was catalyzed in presence of UV irradiation as evidenced by observing a faster increase in the absorbance at wavelengths higher than 700 nm. In addition, the increase in absorbance at wavelengths between 310 and 380 nm was assigned to the formation of Pt²⁺ [32]. As a result, it was inferred that the polymerization of EDOT was accompanied by the reduction of Pt⁴⁺ to Pt²⁺, and then to Pt. The kinetics of the polymerization also affected the morphology of the prepared PtNPs. As revealed in Figure 3, PtNPs with smaller size were obtained under UV irradiation, which can be attributed to the faster nucleation rate of PtNPs.

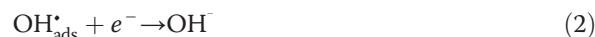
The formation of metallic PtNPs from chloroplatinic acid hydrate was further confirmed by XPS analysis. As revealed in Figure 4, the Pt 4f spectrum consists of a spin-split double, i.e., Pt 4f_{7/2} (71.4 eV) and 4f_{5/2} (74.9 eV), which is consistent with the presence of metallic oxidation state of Pt [33].

Figure 5a shows the SEM image of the PEDOT-PtNPs/SPC electrode. As revealed in Figure 5a, the morphology of the PEDOT-PtNP composites is sphere-like, uniform in size (*ca.* 100 nm), and well distributed. In addition, the assembly of nanoparticles creates a 3-day

microstructure and nanopores which are beneficial for the diffusion of analytes and would provide highly accessible surface area for the electrocatalytic reaction. Furthermore, from the EDX result, as shown in Figure 5b, the existence of signals for O, S, and Pt confirms that the nanoparticles in Figure 5a belong to PEDOT-PtNP composite.

Sensing behavior of PEDOT-PtNPs/SPC electrode

The electrocatalytic behavior of the PEDOT-PtNPs/SPC electrode towards the electrochemical reduction of H₂O₂ was studied using cyclic voltammetry. Figure 6a,b,c shows the CV responses for the bare SPC, PEDOT/SPC, and PEDOT-PtNPs/SPC electrodes in deaerated 0.1 M phosphate buffer solution (PBS, pH 7.4) containing 0 and 0.1 mM of H₂O₂. In the blank phosphate buffer, no faradic current was detected for all electrodes. However, an obvious change in reduction current density was noticed after the addition of 0.1 mM of H₂O₂ in the case of the PEDOT-PtNPs/SPC electrode, while there were no obvious change in current density for the cases of bare SPC and PEDOT/SPC electrodes. It has been reported that the electroreduction of H₂O₂ on PtNPs involves a rate-limiting chemical step (Equation 1) followed by the electron transfer step (Equation 2) [34]:



As a result, we can infer that the enhanced catalytic current of the sensor can mainly be attributed to the presence of the large number of nanosized PtNPs on the electrode [13]. Furthermore, the effect of the film thickness on the sensing performance was also investigated. Here, the film thickness was controlled by adjusting the times of dropping-coating. As revealed in Figure 6c,d,e, a higher film thickness reduced the current response to the H₂O₂, which could be attributed to a higher diffusion barrier, induced by a thicker film, for the hydroxyl radicals diffuse to the electrode surface.

Amperometric detection of hydrogen peroxide

Figure 7 shows the transient current densities at various H₂O₂ concentrations. The current density responses of the PEDOT-PtNPs/SPC electrode as a function of the H₂O₂ concentration, with a sampling time of 200 s at each concentration level, are measured and shown in Figure 7. The current density increases linearly with the increased H₂O₂ concentration up to 6 mM. The sensitivity and detection limit (*S* / *N* = 3) for the PEDOT-PtNPs/SPC electrode are 19.29 mA cm⁻² M⁻¹ and of 1.6 μM, respectively.

Conclusions

The PEDOT-PtNP composite was successfully synthesized in one-pot fashion *via* a novel photochemical

method, and its application for the detection of H₂O₂ was investigated. The polymerization of EODT accompanied with the formation of PtNPs was confirmed by SEM, TEM, UV-vis, and XPS. In addition, as revealed from the TEM results, the PtNPs were formed and embedded in the nanosized PEDOT, indicating the formation of PEDOT-PtNP composite. As compared with the bare SPC and PEDOT/SPC electrodes, the electrocatalytic activities of PEDOT towards H₂O₂ were enhanced by incorporating PtNPs. A linear relationship could be obtained between the current density and the concentration of H₂O₂ up to 6 mM, suggesting the successful fabrication of a sensor for the detection of H₂O₂ in the concentration range of our interest. The sensitivity of the sensor was determined to be 19.29 mA cm⁻² M⁻¹, and the limit of detection (LOD, with S / N = 3) was 1.6 μM. The response time for reaching steady-state current (t₉₅) was 30 to 40 s. Although the conditions for the photochemical reduction of PtNPs were not optimized, the low LOD (approximately 1.6 μM) in this study renders the PEDOT-PtNP electrode attractive for the determination of H₂O₂.

Competing interests

The authors declare that they have no competing interests.

Author's contributions

Y-HL and C-YL designed the photosynthesis experiments. L-CC and H-NW carried out the photosynthesis experiments. L-CC carried out sensing experiments and drafted the manuscript. SEM and XPS were carried out by C-WH. K-CH supervised the project and was responsible for the accuracy of the data reported. All of the authors discussed and analyzed the data. All authors read and approved the final manuscript.

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