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The electrochemiluminescence of luminol on titania nanotubes functionalised indium tin oxide glass for flow injection analysis



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ABSTRACT

The titania nanotubes (TiNTs) had been immobilised onto the indium tin oxide (ITO) coated glass to intensify the electrochemiluminescence (ECL) of luminol. The morphology, structure and properties such as specific surface area and transmittance of synthesised TiNTs were characterised. The results indicated that the TiNTs was several hundred nanometres in length with the diameter of 20 nm. In flow injection analysis (FIA) mode, the TiNTs dramatically enhanced the ECL emission of luminol for about 25 multiple, meanwhile decreased the requirement of buffer pH and exciting potential. The ECL emission of luminol on functionalised ITO electrode has sensitive response toward hydrogen peroxide, and extraordinarily responsive toward the antioxidant. Under the optimal conditions, the ECL emission exhibited a linear response within the concentration range from 0.1 mg L⁻¹ to 30 mg L⁻¹ and an absolute detection limit of 1.65 × 10⁻¹⁰ g of resveratrol. The gross antioxidant activity of blueberry and kiwi were determined with satisfactory recoveries.

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1. Introduction

The electrochemiluminescence (ECL) has progressed in the past decades as a powerful analytical technique [1–3]. It has the high signal-to-noise ratio thus to bring in the high sensitivity; also is in better controllable and reproducible in comparison with the chemiluminescence (CL). Also, the ECL requires simple instrument, using an electrochemical workstation coupled with a photomultiplier tube to record the signal. This is its remarkable dominance than those photo induced luminescence such as fluorescence where the light source was required. Meanwhile, it needs little detecting consumption and easy operation. Lots of its applications have been explored in the fields of environment, food, immunology, biology and clinical diagnosis *etc.* [4–7]. Luminol is one of the most important and mature species of the luminescent reagents owing to its high luminescent efficiency, feasibility of derivation, and cheap cost [8,9]. However, the ECL of luminol often requires the conditions of alkaline media (up to pH 13.0) and high exciting potential (generally 1.5 V), which limits its bio-related applications [1].

Since the first report on the ECL of silicon quantum-dots (SiQDs), semiconductor nano-crystals (S-NCs) have been widely

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studied [10]. As a type of S-NCs, nanoscaled TiO₂, has attracted great interests because of its nontoxicity, biocompatibility and environmental safety. It has also been used as an intensifier for ECL sensors and emitters [11–13]. In our previous studies, the efforts have been dedicated to develop various nano-titania materials with different morphology; we have confirmed that the nanoscaled TiO₂ could act as a catalytic material to significantly promote the ECL strength of luminol, meanwhile reducing the requirement of the medium alkalinity and the exciting potential [14–16]. But, it is absolutely that the solid sphere of nano-titania is un-benefit for light transparency especially in the flow-injection analysis (FIA) mode because of its opacity. Currently, several nano-structured TiO₂ materials, such as nanotubes, arrayed nanotubes, nanobelts, nanowires and nanorods, have been synthesised for a lot of important research fields [17–20] because of their better performance than artless titania nano-particles (TiO₂NPs). We have also synthesised the hollow-shells of nano-titania (TNHSs), and affirmed its better efficiency than TiO₂NPs by improved light transparency [21], reaching the level of those nano-composites, such as nano-titania hybrids of Au or AuAg [14,15]. Nevertheless, the synthesis of TNHSs is very complex, difficult and low yielding.

Of these new nano-structured materials, greater attention has focused on the applications of titania nanotubes (TiNTs) because of its nanosized channels, high ion-exchange capability, high surface area, stable mechanical and chemical properties, biocompatibility, good electronic conductivity, catalytic and optical properties [22,23].

It has been used in biosensors, biomedicines, ion-exchange applications, lithium batteries, solar cells, wastewater treatment, photocatalysis and catalytic oxidation [20,24–26]. The methods of template deposition, anodic oxidation or hydrothermal reaction have been applied to prepare TiNTs. The easy and controllable hydrothermal method is the simplest route for its large scale preparation with low cost [27]. Some researches had exploited the TiNTs in the development of several chem-/bio-sensors [28–31].

In our previous works, we built a flow injection ECL analytical (ECL-FIA) system [32,33]. It has many advantages over conventional ECL equipment, including high analytical efficiency, small sample requirement and good reproducibility. In this system, indium tin oxide (ITO)-coated glass is used as the working electrode to construct the flow ECL cell. It is beneficial to the simplicity and low price of the cell, but lacks the sensitivity due to certain limitations of the ITO glass itself. Also, our researches have proved that the ECL of luminol very closely related to reactive oxygen species (ROs). Herein, we

functionalised the ITO with TiNTs to improve its characters. After optimisation of the parameters of the ECL measurements, about 25 times the ECL emission compared to that on bare ITO electrode was obtained. With low concentrations of luminol and low pH, the ECL signal exhibited a sensitive response toward ROs or antioxidants, such as hydrogen peroxide or resveratrol.

Oxidative stress (OS), one negative effect in life bodies caused by excessive oxygen free radicals, leads to aging and diseases via damaging cell components as lipids, membrane, protein or DNA [34–36]. Its evaluation is very important in clinical diagnosis and pathologic research. Apak and coworkers recently reported a colourimetric sensor for the simultaneous detection of ROs and antioxidant (AOX) [37]. Compared to this simple method, our technique has a lot of advantages such as higher sensitivity, wider linear range and lower detection limit. Also, with a very small sampling volume, reached to 2 μL in extreme situations [38,39], our technique is in promising of use for those applications as cell

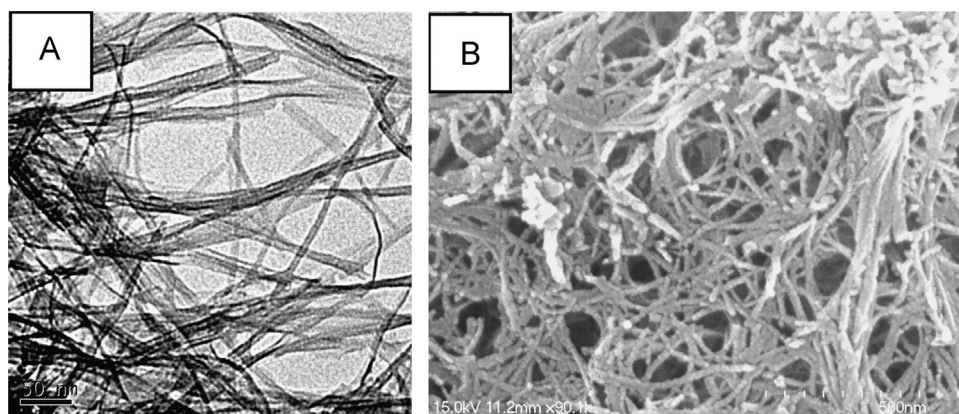


Fig. 1. (A) The TEM image of TiNTs and (B) the SEM image of sintered TiNTs films.

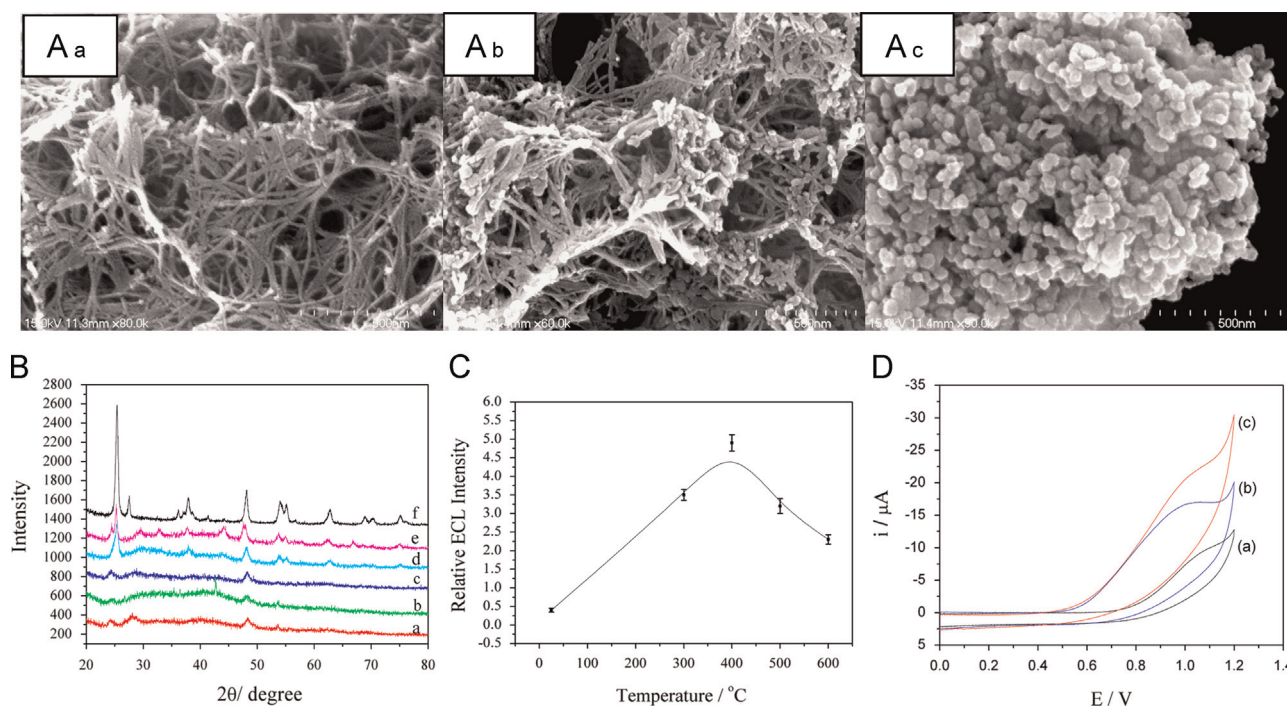


Fig. 2. (A) The SEM images of TiNTs after calcined at (a) 400 °C, (b) 500 °C and (c) 600 °C. (B) The X-ray diffraction of TiNTs at (a) room temperature and after calcined at (b) 200 °C, (c) 300 °C, (d) 400 °C, (e) 500 °C, and (f) the raw material. (C) The effect of calcination temperature for TiNTs on ECL emission. (D) The cyclic voltammograms of $1 \times 10^{-5} \text{ mol L}^{-1}$ luminol on (a) bare ITO electrode, (b) the TiNTs functionalized electrode and (c) after addition of $5 \times 10^{-5} \text{ mol L}^{-1} \text{ H}_2\text{O}_2$ with a scan rate of 0.05 V s^{-1} .

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