



Short communication

## Carboxyl hydrogel particle film as a proton source for electrode surface modification

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## ARTICLE INFO

## Article history:

Received 19 October 2013

Received in revised form 6 November 2013

Accepted 8 November 2013

Available online 15 November 2013

## Keywords:

Cyclic voltammetry

Hydrogel particle

Modified electrode

Cavity electrode

Dissociation of carboxylic acid

## ABSTRACT

Uniformly sized poly(*N*-isopropylacrylamide-co-acrylic acid) (NIPA/AA) hydrogel particles were synthesized as a solid-acid for electrode surface modification. Voltammograms of the NIPA/AA particle film-coated platinum electrodes showed the reduction of hydrogen ions which were supplied from the dissociation of carboxyl groups of the absorbed NIPA/AA particles. The reduction peak potentials at the particle film-coated cavity platinum electrode were observed around  $-0.38$  V. Compared with that observed at flat platinum electrode, the peak potentials were shifted in the positive direction by  $0.19$  V. The peak currents were increased with increase in the amount of immobilized particle. The particle film worked as a source of supply of hydrogen ions for the electrochemical reduction of 1,4-naphthoquinone. The electrode surface pH was controlled to be 4.5 by NIPA/AA particle film in the neutral solution.

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## 1. Introduction

Latex particles have been attracted much interests for electrode surface modification to improve the mechanical, catalytic and electronic properties during the past two decades [1–4]. Polyaniline/polystyrene particle film-coated iron electrodes have exhibited the anti-corrosive properties [5]. Polystyrene/hemoglobin particle film-coated graphite electrodes have showed strong catalytic activity of  $O_2$ ,  $H_2O_2$ ,  $NO_2^-$  and trichloroacetic acid [6]. Particle composed of DNA,  $Fe_3O_4$  and polyacrylate/polystyrene has presented very low detection limit ( $12$  CFU·mL<sup>-1</sup>) for *Salmonella* [7]. Unfortunately, further application of latex particles to electrode surface coating is limited by the geometrical hindrance [8–10] caused by point-to-point contact between the spherical particles and the flat electrode.

Hydrogel particles with configurable shapes and chemistries have garnered considerable attention for improving the drawback of the geometrical hindrance of the electrode [11]. The reason is due to the fact that hydrogel particles are consist highly hydrated networks of cross-linked polymer chains, which possess a considerable degree of flexibility and porosity and thus allow ions and molecules access to the interior space of the particles. These qualities make hydrogel particles particularly attractive candidates for electrode surface modification.

A typical hydrogel particle is poly(*N*-isopropylacrylamide-co-acrylic acid) (NIPA/AA) particle which has been of particular interest due to their stability, versatility, reproducibility, and low cost of production

[12]. Abundant amount of carboxyl groups are present not only on the surface but also inside of the particle. Therefore, under lack of buffer solution, the particle film can be used as a solid-acid to supply protons directly to proton-electron coupled reactions at the electrode surface. In the present report, NIPA/AA particle was synthesized and applied to the cavity platinum (CPt) and flat platinum (FPt) electrode surface modification. Our attention was focused on the effect of the electrode structure on the voltammetric behaviors of the particle. The result was applied to a source of hydrogen ion in the electrochemical reduction of 1,4-naphthoquinone.

## 2. Experimental section

## 2.1. Chemicals

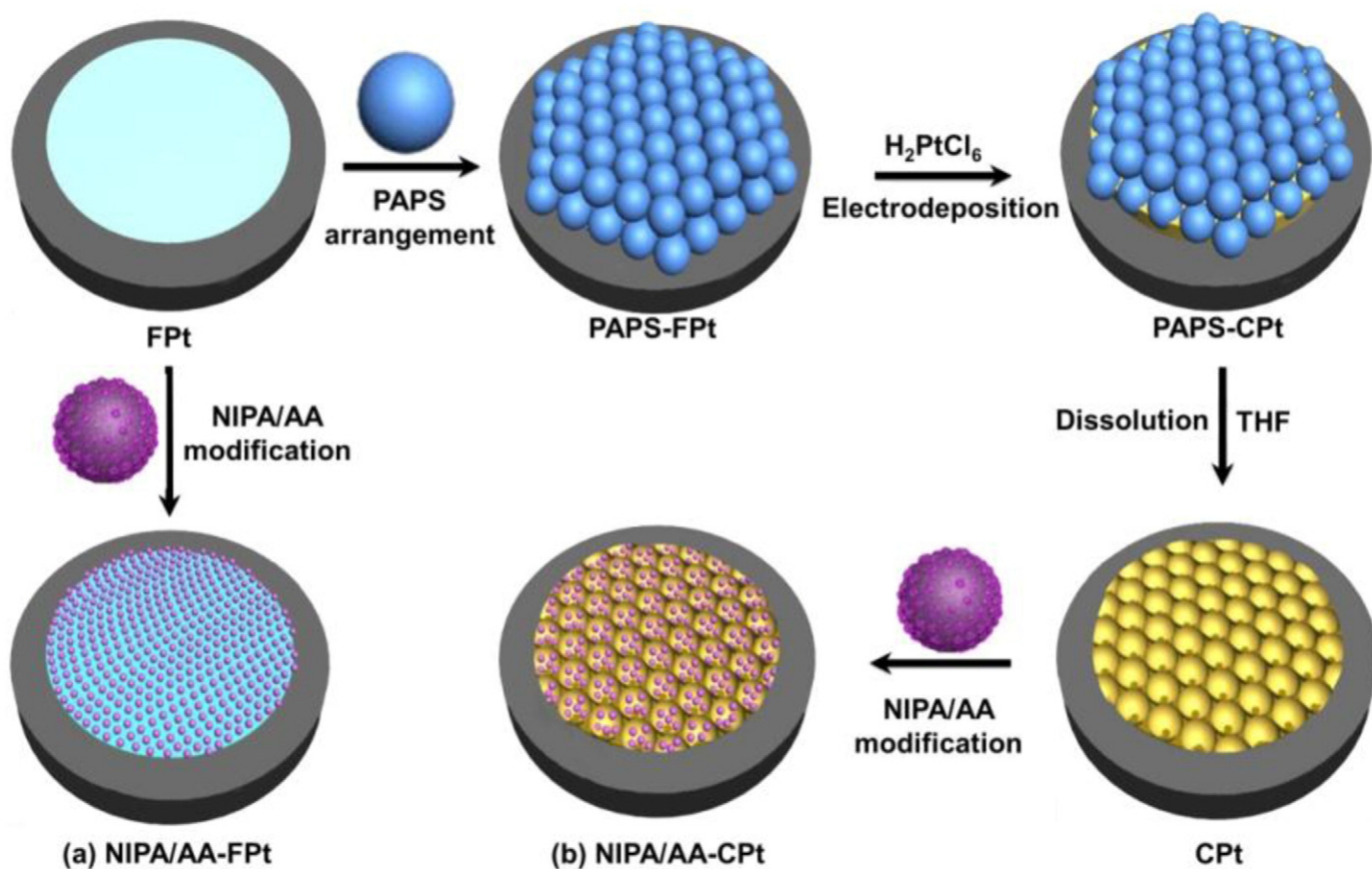
Styrene and acrylic acid were purified by distillation under vacuum. *N*-isopropylacrylamide was purified by recrystallization from a mixture of toluene and hexane (1:1, v/v). *N,N*-methylene-bis-acrylamide (99%; Sigma-Aldrich), poly(*N*-vinylpyrrolidone) (MW 360 kg·mol<sup>-1</sup>; Sigma-Aldrich) and 1,4-naphthoquinone were used as received. The initiators,  $\alpha$ -azoisobutyronitrile and potassium persulfate were purchased from Aladdin Reagents (Shanghai, China). All the other reagents were of reagent grade and used as received. Aqueous solutions were prepared using ultra-pure water with a resistance of 18.2 M $\Omega$ ·cm (Milli-Q, Millipore).

## 2.2. Preparation of particle film-coated electrodes

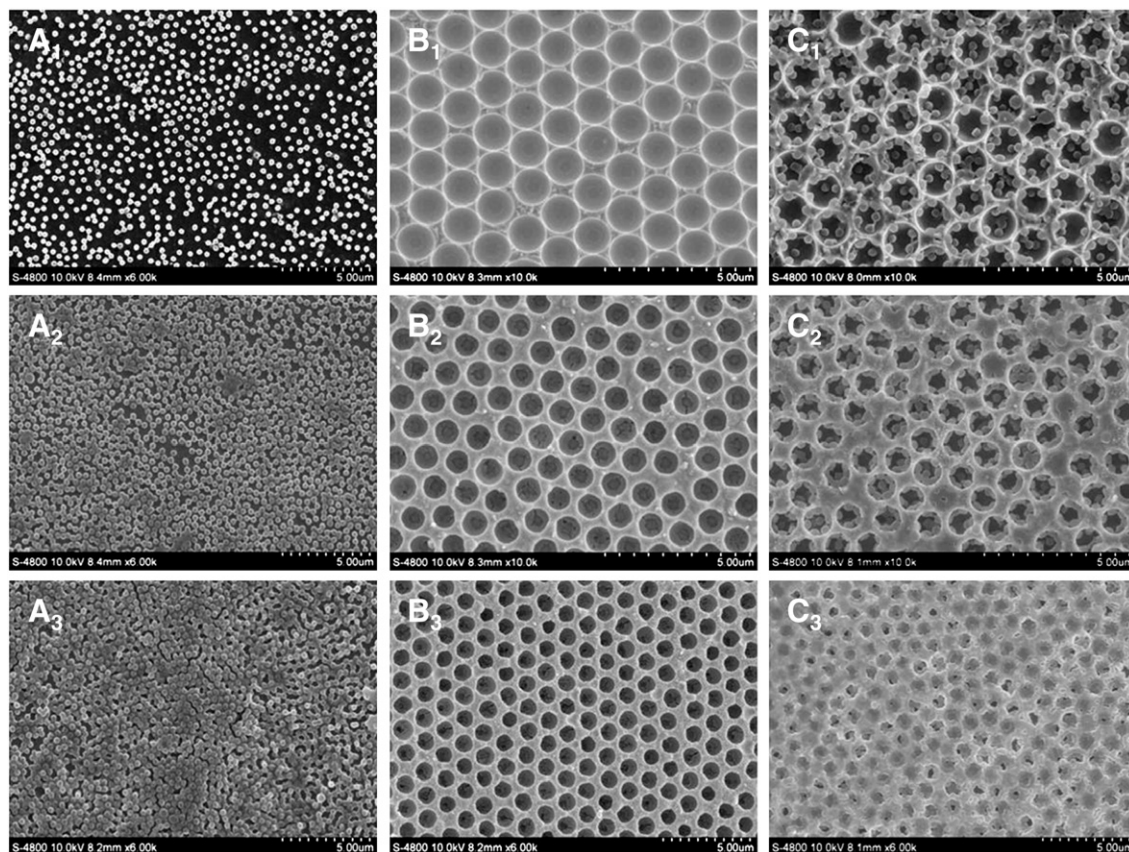
Negative charged polyacrylate/polystyrene (PAPS) particles were prepared by a two-step dispersion copolymerization as described

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**Scheme 1.** Schematic illustration of the procedures for fabricating the NIPA/AA particle film-coated FPt electrodes (a) and CPt electrodes (b).



**Fig. 1.** SEM photographs of NIPA/AA-FPt (A<sub>1</sub>, A<sub>2</sub> and A<sub>3</sub>), bare CPt (B<sub>1</sub>, B<sub>2</sub> and B<sub>3</sub>), and NIPA/AA-CPt (C<sub>1</sub>, C<sub>2</sub> and C<sub>3</sub>) electrodes.

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