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# Investigation of nanoporous carbon supported palladium–zinc nanocomposites as anode catalysts for direct borohydride–hydrogen peroxide fuel cell

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## ARTICLE INFO

### Article history:

Received 8 February 2015

Received in revised form

29 March 2015

Accepted 12 April 2015

Available online 30 April 2015

### Keywords:

Direct borohydride–hydrogen peroxide fuel cell  
Anode electrocatalyst  
Bimetallic catalyst  
Nanoporous carbon  
Palladium–zinc

## ABSTRACT

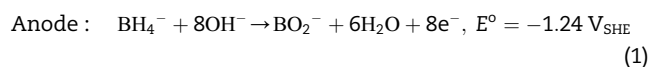
A series of nanoporous carbon supported Pd–Zn catalysts (Pd<sub>x</sub>Zn/NPC, x = 1, 2, 3) have been successfully prepared via impregnation-reduction method and firstly employed as anode catalysts for direct borohydride–hydrogen peroxide fuel cell (DBHFC). The properties of the Pd<sub>x</sub>Zn/NPC electrocatalysts are characterized by X-ray diffraction (XRD), transmission electron microscopy (TEM), cyclic voltammetry (CV), chronoamperometry (CA) and single fuel cell test. The TEM images of Pd<sub>2</sub>Zn/NPC catalyst reveal that the metal particles with a size of approximately 3.1 nm are uniformly dispersed on the nanoporous carbon (NPC). Besides, all the Pd<sub>x</sub>Zn/NPC catalysts exhibit enhanced catalytic activity and stability for BH<sub>4</sub><sup>−</sup> electrooxidation compared to monometallic Pd/NPC in the electrochemical tests. Especially, the Pd<sub>2</sub>Zn/NPC catalyst exhibits the highest electrocatalytic activity toward borohydride oxidation among the four catalysts. Moreover, the DBHFC equipped with the Pd<sub>2</sub>Zn/NPC anode also presents an excellent cell performance with the maximum power density as high as 103.93 mW cm<sup>−2</sup> at 25 °C.

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## Introduction

Sodium borohydride (NaBH<sub>4</sub>), a non-combustible, chemically stable and non-toxic compound, is regarded as one of the promising hydrogen sources of fuel cell with large theoretical specific energy (9296 Wh kg<sup>−1</sup>) and specific capacity (5669 Ah kg<sup>−1</sup>) [1–4]. Thus, the DBHFC that usually utilizes the NaBH<sub>4</sub> in aqueous alkaline medium directly as a fuel has

attracted considerable attention as a good candidate for portable and mobile applications since it eliminates hydrogen storage problems and catalytic poisoning problems [5–9]. Additionally, it has high cell voltage of 3.01 V. The reactions in the DBHFC can be described as follows [10]:

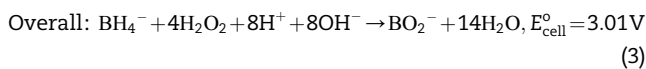
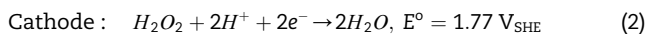


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<http://dx.doi.org/10.1016/j.ijhydene.2015.04.047>

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Although the DBHFC has many advantages over other types of fuel cells, the commercial viability of the DBHFC is confronted with various issues including the high cost of the precious metal-based electrocatalysts and expensive fuel [2]. Anode performance of the DBHFC, as a result of careful selection of the anode catalyst, is crucial to minimize the electrocatalyst cost and improve the fuel utilization. In the past decades, a variety of different materials have been well studied as anode catalysts such as noble metals (Pt, Pd, Au, Ag) [11–13] and transition metals (Co, Ni, Zn) [8,14–16]. Usually, Au anode catalyst is regarded as an effective catalyst for  $\text{BH}_4^-$  oxidation [16,17] because of an eight-electron process of electrooxidation of  $\text{BH}_4^-$  on Au electrode. But Chatenet et al. [18] claimed that the number of electrons released from per  $\text{BH}_4^-$  species is less than 8 at high potentials (>0.3–0.5 V vs. SHE) and Au anode often suffers from low current and power output [19]. Also, the Pt and Pd are the most preferred metals in the literature owing to the fast electrode kinetics of  $\text{BH}_4^-$  on their electrodes [12,20]. However, they have limited storage and high cost, thus increases the total cost of catalysts. In order to reduce the overall use of the noble metals, intense studies have been devoted mainly to investigate the Pt-based alloy catalysts. These catalysts can not only lower the cost of the electrocatalysts to some extent, but also keep or even enhance the catalytic activity of borohydride oxidation [21–25]. Tegou et al. [25] investigated the Pt–Ni and Pt–Co alloys for electrochemical oxidation of borohydride and found that the catalytic activity of the bimetallic catalysts in the kinetic and mixed control potential range were higher than that of pure Pt. Our previous researches also obtained better catalytic activity and stability toward  $\text{BH}_4^-$  oxidation by using carbon supported Pt–Cu, Pt–Sn, Pt–Co and Pt–Zn binary alloys instead of Pt/C [26–29]. Similarly, Pd shows fast electrode kinetics and good power performance in DBHFC, but it is relatively cheap in contrast to Pt. More recently, some studies have focused on the Pd-based alloy catalysts. For instance, Behmenyar et al. [30] found that the carbon supported Pd–Cu bimetallic catalysts have much higher catalytic activity for the direct oxidation of  $\text{BH}_4^-$  than the carbon supported pure nanosized Pd catalyst. de León et al. [10] studied the competition between borohydride oxidation and hydrogen evolution on the Pd–Ir alloys compared with that on pure Pd, and reported that the presence of Ir favors borohydride oxidation rather than hydrogen evolution. Obviously, alloying Pd with a non-noble metal is another effective means to optimize performance and minimize the cost of the catalysts in DBHFC.

As well known, Zn exists widely in the nature and its cost is low. More importantly, it has been claimed as a very effective element to increase the utilization of the borohydride [8,31,32]. However, to the best of our knowledge, the studies concerning nanoporous carbon (NPC) supported Pd–Zn catalysts for borohydride electrooxidation are rarely reported. Based on the above analysis, a series of Pd–Zn bimetallic nanoparticles supported on NPC were prepared in the present paper. The performance of the as-prepared catalysts in

alkaline  $\text{NaBH}_4$  was evaluated in details by using CV, CA and single cell test.

## Experimental

### Synthesis of $\text{Pd}_x\text{Zn}/\text{NPC}$ electrocatalyst

Recent studies at our laboratory have suggested that the NPC prepared by template metal-organic framework-5 (MOF-5) can be a good carrier for anode catalysts in DBHFCs due to its excellent properties such as high specific surface area and large pore volume [33]. The preparation method of the NPC was based on the procedure described in our previous work [33]. Briefly, the MOF-5 ( $[\text{Zn}_4\text{O}(\text{bdc})_3]$ , bdc = 1,4-benzenedicarboxylate) template was prepared by solvothermal method with the optimal synthesis conditions according to the reported strategy [34]. Then the carbon precursor furfuryl alcohol (FA) was infiltrated into the pores of the MOF-5 template via the incipient wetness technique. And the FA/MOF-5 composite was polymerized and converted to a nanoporous structure by carbonization in an Ar atmosphere. Ultimately, the as-prepared sample was treated with HCl aqueous solution (0.1 M) to remove Zn resulting from the decomposition of MOF-5 template. The treated sample was denoted as NPC.

Using conventional impregnation-reduction method, the series of  $\text{Pd}_x\text{Zn}/\text{NPC}$  electrocatalysts with different Pd/Zn molar ratios (1:1, 2:1 and 3:1) were prepared. For instance, the PdZn/NPC electrocatalyst with a Pd/Zn molar ratios of 1/1 was prepared as follows: First, 0.5 mL of 0.1 M  $\text{H}_2\text{PdCl}_4$  solution, 0.5 mL of 0.1 M  $\text{ZnCl}_2$ , 0.28 mL of polyvinyl pyrrolidone (PVP, 1.1 wt%) and 51.8 mg of NPC were added into 100 mL deionized water successively and the mixture was subjected to magnetic stirring for 30 min. The pH was then slowly adjusted to 10 using NaOH prior to adding the  $\text{NaBH}_4$  solution (1 mL, 1 M). Next, the solution was kept under vigorous stirring for 24 h. Finally, the precipitate was filtered, washed, and dried at 80 °C under vacuum overnight. For comparison, Pd/NPC was also prepared by the same method.

### Physical characterization

JEOL JSM-2100F TEM system operating at an accelerating voltage of 200 kV was primarily used for microscopy analysis. X-ray diffraction (XRD) measurements were carried out on a D/Max-3C diffractometer using  $\text{Cu K}\alpha$  radiation ( $\lambda = 1.54056 \text{ \AA}$ ) with a scan rate of  $5^\circ \text{ min}^{-1}$ . The tube voltage was held at 40 kV and tube current was held at 100 mA. Elemental analysis of the as-prepared catalysts was carried out by energy dispersive spectra (EDS) system, which was attached to the JEOL JSM-6360 scanning electron microscopy.

### Electrochemical measurements

Electrochemical tests were performed on a VersaSTAT3 electrochemical workstation (Princeton, America) equipped with a standard three-electrode system. The glassy carbon (GC, 3 mm in diameter) electrode coated with electrocatalyst ink was used as working electrode, Ag/AgCl (saturated KCl) electrode was used as reference electrode and Ni foam with

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