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ORIGINAL PAPER

Facile synthesis of PtCo nanowires with enhanced electrocatalytic performance for ethanol oxidation reaction

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Abstract

Alloying Pt with 3d transition metals can largely promote the electrocatalytic performances of Pt-based nanocatalysts by downshifting the d-band center of Pt and modulating the electronic structure. Here, PtCo nanowire catalysts are obtained by a simple hydrothermal method. Due to the special one-dimensional (1D) nanowire structure and the strong synergistic effect between Pt and Co, the PtCo nanowire catalysts exhibit high electrocatalytic performances for ethanol oxidation reaction (EOR). The mass activity of optimal Pt_1Co_1 nanowires is 3.69 times of pure Pt. It also shows excellent durability and stability, which can sustain high activity after operating more than 500 cycles of electrochemical cycling. Our works point out a direction for the development of Pt-based nanomaterials with potential industrial application.

Keywords 1D nanowires · PtCo alloy · Ethanol oxidation reaction

Introduction

The non-renewability of traditional fossil fuels and the resulting environmental problems became two major obstacles, which limited the social development. The search for clean, renewable, and low-cost energy is an important strategy to solve these problems [1]. As green energy conversion devices, direct ethanol fuel cells (DEFCs) have been widely regarded as one of the promising technologies to attenuate the crisis caused by energy shortage due to their high energy conversion efficiency, low operating temperature, and low pollution emissions [2, 3]. Electrocatalysts are the core components of DEFCs; therefore, the development of efficient, low-cost, and stable electrocatalysts is the key to achieve the goal of the industrialization of DEFCs [4]. At present, platinum (Pt) and its alloys are the most effective electrocatalysts for driving fuel cell reactions. However, these catalysts are expensive and unstable, which make them difficult to get a practical application [5–9]. Therefore, tremendous efforts have been devoted, such as doping transition metals to reduce the content of Pt, to obtain high-performance electrocatalysts. In this regard, the as-obtained Pt-based nanocatalysts show not only better electrocatalytic activity but also high stability, due to the synergistic effect between Pt and nonprecious metal [10–14]. More importantly, the energy of d-band center of Pt can become lower after the incorporation of 3d transition metals, greatly promoting to the improvement of electrocatalytic performances.

In addition to doping a transition metal element, the synthesis of nanomaterials with specific morphology is also one of the effective methods for improving the electrocatalytic performance [15, 16]. At present, various unique morphologies of Pt-based nanomaterials have been successfully designed, such as flower-like [17–19], block [20, 21], film [22, 23], and core-shell structure [24, 25], but most of these structures have the disadvantages of low activity and poor stability [26, 27]. In recent years, one-dimensional (1D) nanowire structures have become a hot research topic, which ascribed to their anisotropy, Ostwald ripening, and various scale combinations [28–30]. The Pt-based nanowire structure has also been synthesized. For example, Xu and coworkers reported the well-defined mesoporous Pt nanowire which was prepared by chemical reduction deposition [31], Chen and coworkers showed a facile chemical co-reaction method for fabricating chain-like Pt-Cu nanowire [32], Wang and coworkers documented the Pt-Pd nanowire which was synthesized by a facile one-pot aqueous approach with the assistance of L-histidine [33], and Shen and coworkers exhibited a facile method for

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preparing trimetallic PtAuCu nanowires [34]. These nanomaterials exhibit excellent performance in terms of water decomposition, alcohol oxidation, and supercapacitance.

PtCo nanowires, as an advanced class of nanomaterials, have been reported. For example, Chen and his coworkers reported the growth of PtCo nanowires on the free-standing carbon foam framework by a two-step process, which could be employed for efficient ethanol oxidation reaction (EOR) [35]. Buay and coworkers reported the fabrication of PtCo nanowires by a direct electrodeposition [36], which was applied for methanol oxidation reaction (MOR). However, lots of efforts have been devoted for fabricating PtCo nanowires toward electro-oxidation reaction; the complexed synthetic method and unsatisfactory electro-oxidation performance still impeded their practical application. Based on the above analysis, we report a class of 1D PtCo nanowire prepared by a simple hydrothermal method. Due to the unique 1D nanowire structure and the synergistic effect of PtCo, the nanomaterials show good ethanol oxidation performance and suggest the potential applicability of such compounds in proton exchange membrane fuel cells.

Experimental section

Chemicals and materials

Chloroplatinic acid (H_2PtCl_6, 99%), cobaltous chloride ($CoCl_2 \cdot 6H_2O$, AR), potassium hydroxide (KOH, AR), *N*,*N*-dimethylformamide (DMF, AR), and ethylene glycol (EG, AR) were all purchased from Sinopharm Chemical Reagent Co. Ltd. (Shanghai, China). All the reagents were used as received without further purification. The water (18 MΩ cm^{-1}) used in all experiments were prepared by passing through an ultra-pure purification system (Aqua Solution).

Synthesis of PtCo nanowire

In the standard synthesis of Pt_1Co_1 nanowire, 0.02 mM of H_2PtCl_6, 4.8 mg of $CoCl_2 \cdot 6H_2O$, and 10 mM of KOH were added into the solution containing 6 mL of DMF and 4 mL of EG. After sonicating for 5 min to make all the samples completely dissolved, the homogeneous solution was transferred to a reaction vessel and heated from room temperature to 170 °C and maintained for another 8 h. After cooling at room temperature, the products were then collected after centrifuged and washed with ethanol and acetone mixture for several times, and the final product was dispersed in 10 mL of deionized water for further use. The preparation of $Pt_1Co_{0.5}$ and $Pt_1Co_{1.5}$ nanowires is similar to the above synthetic method except that the amount of $CoCl_2 \cdot 6H_2O$ is changed to 2.4 mg and 7.2 mg, respectively. The pure Pt nanocatalyst is similar to the above synthetic procedure without the addition of $CoCl_2 \cdot 6H_2O$.

Characterizations

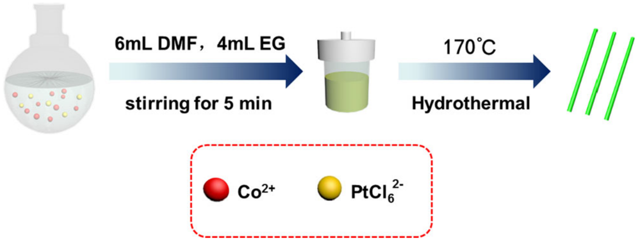
The transmission electron microscope (TEM) was performed with JEM-2100F at 200 kV. X-ray diffraction (XRD: D/MAX–255) was conducted by a Philips advance powder XRD with Cu Ka radiation source (l = 0.1542 nm). X-ray photoelectron spectroscopy (XPS) was conducted on a Thermo Scientific ESCALab 220i-XL electron spectrometer from VG Scientific with 300 W Al Kα X-ray radiation source.

Electrochemical measurements

All the electrochemical tests were performed by using a CHI760E electrochemical station. The glassy carbon electrode (GCE) was selected as the working electrode, which needed to be polished everytime before electrochemical measurements. To prepare the catalyst-coated working electrode, 5.1×10^{-3} mmol of catalyst was dispersed in a mixture containing 4.95 mL of isopropanol and 50 μL of Nafion (5%) to generate a homogeneous dispersion (0.20 mg Pt/mL). Then, 10 μL of catalyst inks was deposited on a GCE to obtain the catalyst-modified working electrode, and the mass of Pt loaded on the surface of GCE is 2 μg. The cyclic voltammetry (CV) for EOR was conducted in 1 M KOH + 1 M ethanol solution with a rate of 50 mV s^{-1}. The continuous CV of 500 cycles and chronoamperometry (CA) measurement at the potential of 0.8 V (vs RHE) for 1 h have also been conducted to evaluate their durability. All electrochemical experiments were performed at room temperature. For comparison, the pure Pt catalyst was used as the reference catalysts.

Results and discussion

We synthesized PtCo nanowires using a simple hydrothermal method as described in Scheme 1. In order to study the morphology and structural characteristics of the material in detail, we employed the transmission electron microscope (TEM), X-ray diffraction (XRD), and X-ray photoelectron spectroscopy (XPS) to characterize the structural characteristics of these compounds. As shown in Fig. 1a, Pt_1Co_1 nanomaterials have mutually interlaced network structure, which is helpful to facilitate the mobility of electron. These network structures are composed of many ultrathin uniform nanowires with a diameter of



Scheme 1 The synthetic schematic illustration of PtCo NWs

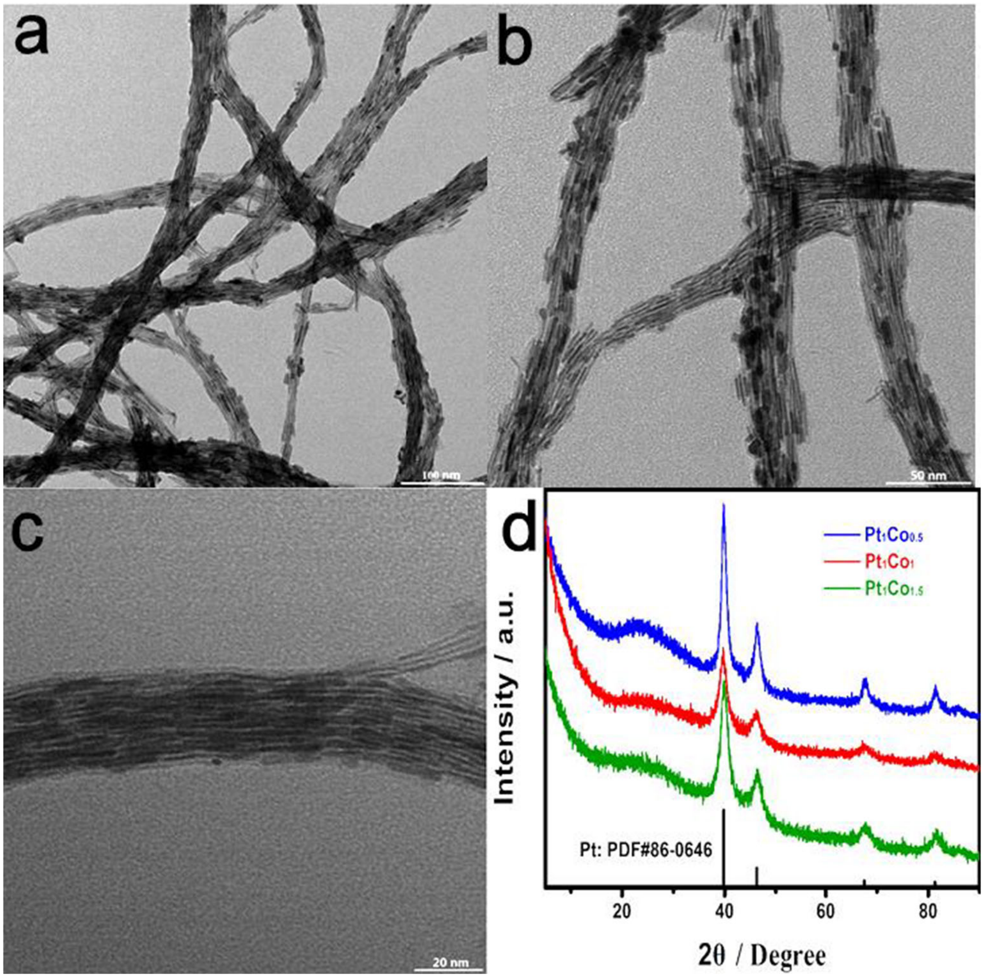


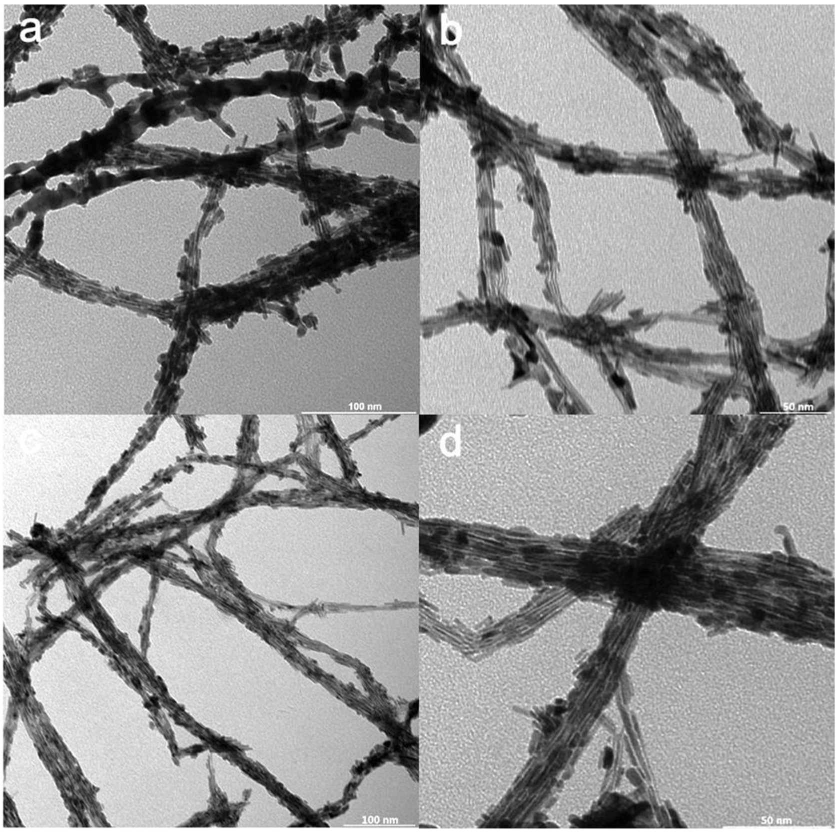
Fig. 1 **a**–**c** TEM images of 1D Pt_1Co_1 nanowires with different magnifications. **d** The PXRD patterns of 1D $Pt_1Co_{0.5}$ nanowires, Pt_1Co_1 nanowires, and $Pt_1Co_{1.5}$ nanowires

20 nm. As seen in Fig. 1b, each nanowire is consisted of ultrathin nanowires with rough surface, which can expose more surface active areas. A detailed observation in Fig. 1c indicated that the surface of the Pt_1Co_1 nanowires contained many pores, which were beneficial for the adsorption and diffusion of intermediates involved in EOR [37]. We used powder X-ray diffraction (PXRD) to characterize the PtCo alloy. As shown in Fig. 1d, the four diffraction peaks of Pt_1Co_1 nanowires are indexed to Pt (PDF#86-0646). However, the XRD patterns of Pt_1Co_1 nanowires can also display a slight shift to high angle in comparison with the standard, due to the incorporation of Co atoms into Pt, indicating the successful construction of PtCo alloy nanocatalysts [38].

In order to witness that the strategy is universally applicable, we have therefore attempted to construct PtCo nanocatalysts with different chemical compositions by adjusting the feed ratio of the reactants. As shown in Fig. 2a, c, the $Pt_1Co_{0.5}$ and $Pt_1Co_{1.5}$ nanowires were successfully prepared using the same synthetic method. We also found that the surface of the $Pt_1Co_{0.5}$ and $Pt_1Co_{1.5}$ nanowires was embedded with ultrafine nanoparticles and the diameters of the nanowires are about 15 nm. After a further observation, it is clearly found that the surrounding of the nanowires consists of some small-sized nanowires, indicating that the Pt_1Co_1 with a suitable ratio is able to maintain the full structure compared with $Pt_1Co_{0.5}$ and $Pt_1Co_{1.5}$ (Fig. 2b, d). And the PXRD patterns of 1D $Pt_1Co_{0.5}$ and $Pt_1Co_{1.5}$ nanowires exhibited a slight high-angle shift, indicating the formation of PtCo alloy (Fig. 1d). These results have also proved that this synthesis strategy could be widely applied to the synthesis of PtCo nanowire catalyst with different chemical compositions [39].

X-ray photoelectron spectroscopy (XPS) was used to study the electronic state and surface chemical components. The XPS spectrum was corrected using C 1s (284.5 eV) as a standard. As shown in Fig. 3a, the binding energies of $4f_{5/2}$ and $4f_{7/2}$ of Pt^0 peaks are 75.30 eV and 72.01 eV, respectively, and the spin-orbit splitting energy between them is 3.29 eV. In addition, the peaks appearing at 73.16 eV and 76.44 eV are assigned to Pt^{2+} [40]. According to the intensity ratio of Pt^0/Pt^{2+}, Pt in PtCo nanowires mainly exists in the form of zero valence state. As shown in Fig. 3b, we can easily see that the two apparent peaks were located at the binding energies of 796.69 and 780.70 eV, which corresponded to the Co $2p_{1/2}$ and Co $2p_{3/2}$ of Co^{2+} [39–41], respectively. The other two

Fig. 2 TEM images of **a**, **b** 1D $Pt_1Co_{0.5}$ nanowires and **c**, **d** 1D $Pt_1Co_{1.5}$ nanowires with different magnifications



peaks at the binding energies of 802.80 and 785.90 eV were ascribed to their corresponding satellite peaks.

Since the synthesized PtCo nanowires have a porous nanostructure, an aspect ratio, and the strong synergistic effect of PtCo, this material may show a large application potential in liquid fuel oxidation. At the same time, because ethanol has the advantages of flammability, low toxicity, and renewability in biomass raw materials, we chose EOR as a model to evaluate the electrocatalytic oxidation performance of PtCo nanowires. First, we evaluated the electrocatalytic activity of the synthesized catalyst by cyclic voltammetry (CV) at a scanning speed of 50 mV s^{-1} in a solution containing 1 M KOH and 1 M ethanol. As shown in Fig. 4a, the CV curves of the PtCo nanowires show a distinct peak in both the positive sweep and the reverse sweep. The peak appearing in the positive sweep is due to the oxidation of ethanol adsorbed on the surface of the Pt catalyst, while the peak at the backward sweeping section is produced by the further oxidation of the intermediate produced by the oxidation of ethanol [42]. Therefore, for the EOR, we usually calculate the catalytic activity of the flat-valley catalyst based on the oxidation peak current density of the positive sweep. Therefore, we can clearly see that Pt_1Co_1 nanowires have the highest

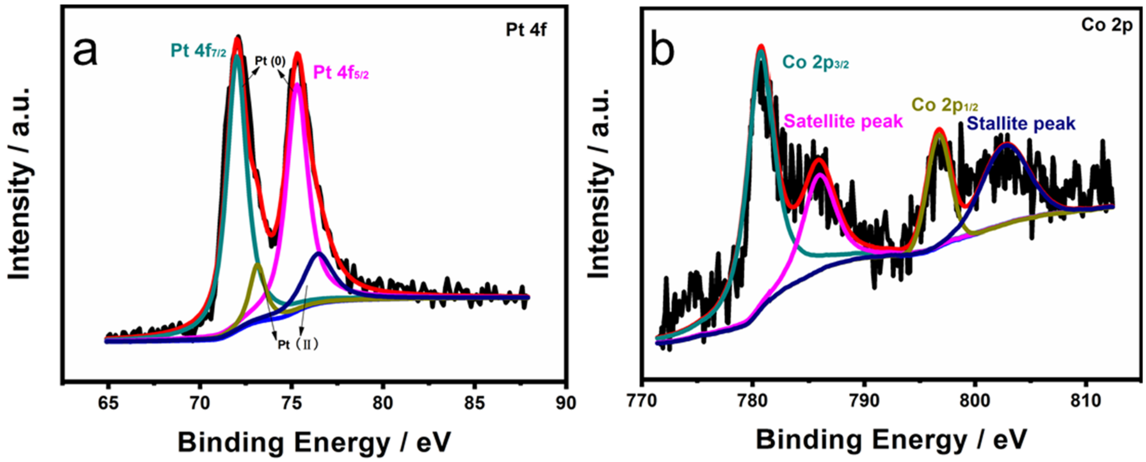


Fig. 3 Deconvolutions of **a** Pt 4f and **b** Co 2p XPS in 1D Pt_1Co_1 nanowires

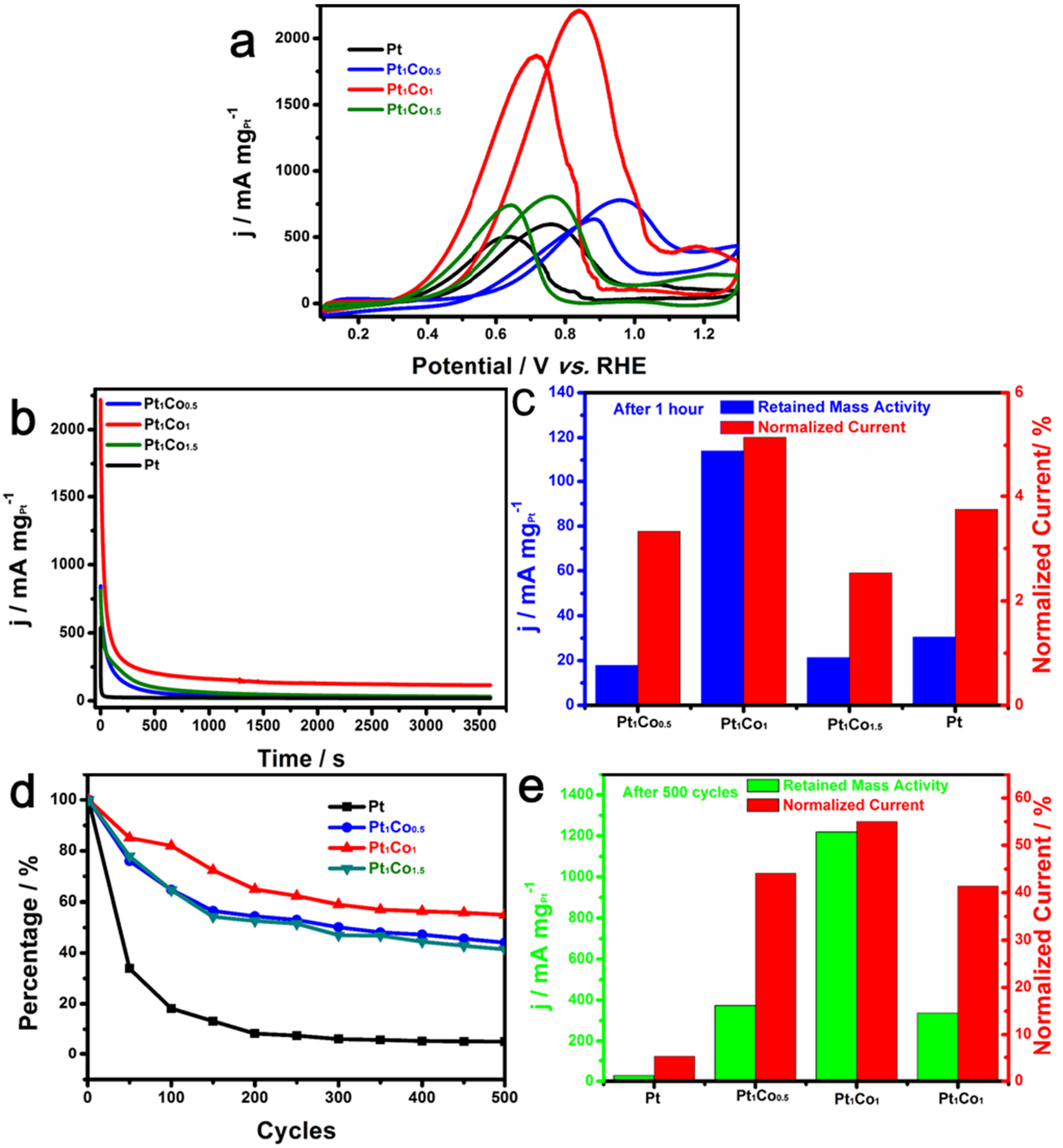


Fig. 4 **a** CV curves of pure Pt, $Pt_1Co_{0.5}$, Pt_1Co_1, and $Pt_1Co_{1.5}$ toward EGOR. **b** CA curves of pure Pt, $Pt_1Co_{0.5}$, Pt_1Co_1, and $Pt_1Co_{1.5}$ for continuous 1 h (at 0.8 V vs RHE). **c** Retained mass activities and normalized current densities of all the samples. **d** Long-term stability comparison of pure Pt, $Pt_1Co_{0.5}$, Pt_1Co_1, and $Pt_1Co_{1.5}$ for continuous CVs of 500 cycles. **e** The retained mass activities and normalized current densities of all the samples after continuous CVs of 500 cycles

electrocatalytic activity of 2203 mA mg^{-1}, followed by $Pt_1Co_{1.5}$ (806 mA mg^{-1}) and $Pt_1Co_{0.5}$ (782 mA mg^{-1}). Pure Pt has the worst electrocatalytic performance (597 mA mg^{-1}). The great enhancement in EOR activity of 1D PtCo nanowires is ascribed to the porous nanowire structure, strong synergistic effect, and modulated electronic structure.

In order to study the electrocatalytic performance of PtCo nanowires in detail, we used the CA test to study the stability of the catalysts. As shown in Fig. 4b, c, the four groups of electrodes showed a large current drop at the initial stage, and then gradually reached to a steady state. The reason why the current density drops significantly during the scanning process can be attributed to the formation of Pt oxide or the intermediates formed in the oxidation process of ethanol, which occupy the active site of the Pt surface and result in the activity decrease [43]. Figure 4c shows the retained mass activities and normalized current percentages of different catalysts after 3600 s. As seen in Fig. 4c, the Pt_1Co_1 nanowires can maintain the mass activity and normalized current percentage of 114.12 mAmg^{-1} and 5.15%, respectively, both of which are much higher than pure Pt (17.9 mAmg^{-1} and 3.75%), $Pt_1Co_{1.5}$ (21.4 mAmg^{-1} and 2.53%), and $Pt_1Co_{0.5}$ (21.4 mAmg^{-1} and 3.33%), indicating the best long-term durability of Pt_1Co_1. To further evaluate the stability of the PtCo alloy and the pure Pt, the continuous CV cycling of 500 cycles has been performed. We took the first circle, the 50th circle, the 100th circle, the 150th circle, the 200th circle, and so on (Fig. 4d). The maximum peak current density of the 500th circle is plotted. The result can be apparently showed in Fig. 4d, where the Pt_1Co_1 showed the highest current density after 500 cycles, followed by $Pt_1Co_{0.5}$ nanowires, $Pt_1Co_{1.5}$ nanowires, and Pt. From Fig. 4e, it can also be more apparently to understand that the residual current density and normalized current of Pt_1Co_1 nanowires are 1218.8 $mAmg^{-1}$ and 55%, respectively, which are significantly higher than $Pt_1Co_{0.5}$ nanowires (373.09 $mAmg^{-1}$ and 51.8%), $Pt_1Co_{1.5}$ (336.83 $mAmg^{-1}$ and 44.1%), and pure Pt (27.68 $mAmg^{-1}$ and 41.4%). Therefore, the electrocatalytic performance of Pt_1Co_1 nanowires is remarkably better than the other two PtCo alloys and pure Pt. In general, the reason for the apparent increase in Pt_1Co_1 catalyzed ethanol oxidation performance may be due to the relatively uniform nanowire structure of this material and the synergistic effect between Pt and Co [44–46].

Conclusions

In general, we have prepared a class of composition-tunable PtCo alloy nanocatalysts with the unique 1D nanowire structure by a simple hydrothermal method. Compared with the pure Pt, the incorporation of Co not only decreases the cost of Pt, but also increases the electrocatalytic performance for EOR. The synthesized Pt_1Co_1 nanowires exhibit high performance due to uniform structure and possible synergistic effects and electronic effects between Pt and Co, whose mass activity was 3.69 times higher than that of pure Pt. In addition, all of the PtCo nanowires also possessed good long-term stability, and the optimal Pt_1Co_1 nanowires could sustain 55% electrocatalytic activity of the initial value after 500 cycles of CV cycling, indicating that we have prepared a new type of nanowire electrocatalyst with excellent catalytic performance for ethanol oxidation.

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