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Enhanced catalytic performance of a Pt-xCeO$_2$/Graphene catalyst for DMFCs by adjusting the crystal-plane and shape of nanoscale ceria

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Oxygen storage capacity is influenced by the morphology and crystal-plane(s) of CeO$_2$, which can thus affect the ability of this material to oxidise carbon monoxide. To investigate the effect of different morphologies/crystal-planes of CeO$_2$ on the electrocatalytic performance of DMFCs (Direct Methanol Fuel cell), three different types of CeO$_2$ nanocrystals with different crystal-planes were synthesised and later assembled into Pt-xCeO$_2$/Graphene composites with graphene and Pt nanoparticles as the electrocatalyst for DMFCs. According to the HRTEM images, the original morphology and crystal-plane structures of CeO$_2$ are essentially maintained in the three types of Pt-xCeO$_2$/Graphene composite catalysts investigated in this work. The catalytic performance of the Pt-xCeO$_2$/Graphene composites for methanol electrocatalytic oxidation was investigated by a series of electrochemical measurements. Compared with the other catalysts, Pt-rCeO$_2$/Graphene demonstrates superior catalytic activity (onset potential: 0.15 V) and the strongest resistance to poisoning by carbonaceous species ($I_{\text{f}}/I_{\text{a}}$: 2.11). The results of H$_2$-TPR shows that rCeO$_2$ with the [110] facet has the best surface reducibility among the xCeO$_2$ with different facets being investigated, which provides a rationale for the superior performance of the Pt-rCeO$_2$/Graphene catalyst. This study indicates that metallic oxides with a suitable crystal plane and shape can effectively enhance the electrocatalytic performance of Pt-based catalysts for methanol electrooxidation.

Introduction

Because of their high energy density, low operating cost, compact design, and reliance on a liquid fuel (methanol), direct methanol fuel cells (DMFCs) exhibit great promise for practical application in transportation and portable devices.\textsuperscript{1} Pt-based catalysts, which are the best catalysts in anodic electrocatalysis of DMFCs\textsuperscript{2}, have been limited by their low utilization and high cost\textsuperscript{3}. Moreover, these catalysts are easily poisoned by reaction intermediates such as carbon monoxide$^4$. To overcome such defects, efforts have been devoted to developing Pt-based alloys such as Pt-Ru, Pt-Ni, Pt-Au, etc. for use as catalysts.$^5$ Additionally, researchers have found that metal oxides can efficiently promote electrocatalytic and anti-poisoning properties of Pt-based catalysts. In this context, Pt-based metal oxides composites, such as Pt/CeO$_2$, Pt/MnO$_2$, Pt/Fe$_2$O$_3$, and Pt/TiO$_2$, have been shown to improve the electrocatalytic performance of Pt-based nanoparticles while demonstrating significant tolerance to CO poisoning. Among the metal oxides, CeO$_2$ is of particular interest because of its high activity during the oxidation of carbon monoxide.$^6, 9$ Recently, Chu et al.$^{10}$ reported that the composite derived from loading Pt/CeO$_2$ onto a carbon-based material showed high activity for methanol electrooxidation and significant tolerance to CO poisoning.

In previous research$^{6, 8-10}$, all of the Pt-CeO$_2$/carbon-based materials were evaluated to achieve the greatest electrocatalytic activity either by i) tuning the scale of CeO$_2$ in the composite material, ii) changing the carbon-based supports, or iii) improving the dispersion of Pt nanoparticles. In fact, because of its incredible physicochemical properties such as high surface area, sizable surface defects, and high oxygen storage capacity (i.e., Ce$^{3+}$/Ce$^{4+}$ redox sites)$^{11}$, the presence of ceria in Pt/CeO$_2$-based composites mostly acts as a promoter that improves the oxidation of CO to CO$_2$ via the lattice oxygen species.$^{12}$ This unique redox property is usually described in terms of a material’s oxygen storage capacity (OSC)$^{11-13}$.\textsuperscript{11-13}
It is well known that the redox features of ceria as described by its OSC are largely influenced by its morphology and depends on the exposed crystal plane.\textsuperscript{13} Ceria with different morphologies and crystal-planes will exhibit different reducibilities, which could profoundly affect its capacity to oxidize carbon monoxide\textsuperscript{14-16}. For example, Si et al.\textsuperscript{14} studied the shape and crystal-plane effects of nanoscale ceria on the activity of Au-CeO\textsubscript{2} catalysts for the water-gas shift reaction and found that the rod-shaped CeO\textsubscript{2} nanocrystals enclosed by the (110) and (100) planes were the most active for carbon monoxide oxidation. In this context, a designed surface morphology/crystal plane CeO\textsubscript{2} with high reducibility is especially important for improving the activity and durability of catalysts for DMFCs.

Owing to its unique properties such as a high surface area and high electrical conductance, graphene (G) is an attractive material to be used as a conductive support in DMFCs.\textsuperscript{5,9,17} Here, we have successfully synthesised three types of CeO\textsubscript{2} nanocrystals with different morphologies/lattice planes: rod-shaped CeO\textsubscript{2} nanocrystals with the (110) and (100) crystal planes, cube-shaped CeO\textsubscript{2} nanocrystals with the (100) crystal plane, and polyhedron-shaped CeO\textsubscript{2} nanocrystals with the (111) and (100) crystal planes. The nanocrystals are assembled with graphene and Pt nanoparticles to form the Pt-xCeO\textsubscript{2}/G composite catalyst, where xCeO\textsubscript{2} indicates the shape of the cerium oxide species. Specifically, rCeO\textsubscript{2} indicates a rod-shaped CeO\textsubscript{2}, cCeO\textsubscript{2} indicates a cube-shaped CeO\textsubscript{2}, and pCeO\textsubscript{2} indicates a polyhedra-shaped CeO\textsubscript{2}. The composite catalyst was used to investigate the effect(s) of the different morphologies/lattice planes of CeO\textsubscript{2} on the electrocatalytic properties of DMFCs. The results indicate that the rod-shaped CeO\textsubscript{2} enclosed by the (110) and (100) facets can greatly enhance the catalyst’s activity for methanol electrooxidation. Moreover, relative to the other CeO\textsubscript{2} species investigated, rod-shaped CeO\textsubscript{2} has a higher resistance to poisoning by carbonaceous species, which we attribute to a higher concentration of oxygen vacancies on the CeO\textsubscript{2} (110) facet.

Experimental Section

Material

All chemicals were of analytical grade and used as received without further purification. Potassium permanganate (K\textsubscript{2}MnO\textsubscript{4}), phoshoric acid (H\textsubscript{3}PO\textsubscript{4}), sulfuric acid (H\textsubscript{2}SO\textsubscript{4}), absolute ethanol (C\textsubscript{2}H\textsubscript{5}OH), sodium hydroxide (NaOH) and ethylene glycol (EG) were purchased from Tianjing Chemical Reagents Limited Cooperation (Tianjing, China). Cerium (III) nitrate hexahydrate (Ce(NO\textsubscript{3})\textsubscript{3}-6H\textsubscript{2}O), hydrogen peroxide (H\textsubscript{2}O\textsubscript{2}) and Nafion\textsuperscript{®} were purchased from Sinopharm Chemical Reagent Limited Cooperation. Other reagents were purchased from Aladdin Industrial Corporation.

Synthesis of graphene oxide

Graphene oxide (GO) was synthesised by a slightly modified Hummers method.\textsuperscript{18} Briefly, 3.0 g of flake graphite was added to a 9:1 mixture of H\textsubscript{2}SO\textsubscript{4}/H\textsubscript{3}PO\textsubscript{4} (360/40 mL). Then, 18.0 g of K\textsubscript{2}MnO\textsubscript{4} was added to the above solution while stirring. The reaction was then heated to 50°C and stirred for 24 h. Once the solution was cooled to room temperature it was poured onto ice (approximately 400 mL). A 30% H\textsubscript{2}SO\textsubscript{4} solution was then added dropwise into the cooled solution until the colour tuned brown. The obtained product was collected by centrifugation, washed repeatedly with deionized water and ethanol and dried in vacuum at 60°C for 10 h.

Synthesis of different shaped CeO\textsubscript{2} nanoparticles

Schematic diagram for the shape-selective synthesis of CeO\textsubscript{2} nanopolyhedra, nanocubes and nanorods was shown in Fig. S1. The synthesis of rod-shaped CeO\textsubscript{2} followed the procedure of Mai et al.\textsuperscript{19} Briefly, Ce(NO\textsubscript{3})\textsubscript{3}-6H\textsubscript{2}O (1.736 g) was dissolved in deionized water (40 mL), and in another solution NaOH (19.2 g) was also dissolved in deionized water (40 mL). Then, these two solutions were mixed in a Teflon bottle and stirred for 30 min at room temperature until a milky slurry was formed. Subsequently, the Teflon bottle was tightly sealed and hydrothermally treated in a stainless-steel autoclave at 100°C for 24 h. After cooling, the obtained white precipitate was collected by centrifugation, washed with deionized water, and dried at 60°C in air overnight. The synthetic procedure for cube-shaped CeO\textsubscript{2} was similar to the synthesis the rod-shaped CeO\textsubscript{2} except that the hydrothermal treatment temperature was set to 180°C. Likewise, the synthetic procedure for polyhedra-shaped CeO\textsubscript{2} was identical to that employed for rod-shaped CeO\textsubscript{2} except that the weight of NaOH was 0.32 g.

Synthesis of the Pt-xCeO\textsubscript{2}/Graphene catalyst

30 mg of the obtained xCeO\textsubscript{2}, 60 mg of graphene oxide and 20 mg of polyvinyl pyrrolidone (PVP) was dispersed into 18 mL of distilled water and ultrasonicated for 0.5 h. Then, 2 mL of 0.05 M aqueous solution of chloroplatinic acid (H\textsubscript{2}PtCl\textsubscript{6}) was added, stirred for 1 h, and then 60 mg of NaBH\textsubscript{4} was added. After the mixture was stirred for 2 h, it was then heated to 60°C and stirred for an additional 30 min. After the mixture was cooled to room temperature, the obtained product (i.e., the Pt-xCeO\textsubscript{2}/Graphene electrocatalyst) was collected by centrifugation, washed with deionized water, and dried at 60°C in air overnight. The synthetic procedure is shown in Scheme 1.

Scheme1. Schematic synthetic protocol for Pt-rCeO\textsubscript{2}/Graphene, Pt-cCeO\textsubscript{2}/Graphene, Pt-pCeO\textsubscript{2}/Graphene composite materials.
operated at 200 kV), and the energy dispersive X-ray (EDS) spectrogram was recorded from the same FESEM. The powder X-ray diffraction (XRD; Brucker, D8-Advance) with Cu-Kα radiation (λ=0.15406 nm) was used to characterise the composition and crystal structure of the samples. The H2-TPR experiment was performed using a Micromeritics Chemisorb 2750 TPD/TRP system. Typically, 50 mg of xCeO2 was loaded into a U-shaped quartz tube. After pre-treatment for 1 h at 250°C in a flowing 20% O2/N2 mixture (25 mL/min), the samples were cooled to room temperature under flowing N2. The samples were then heated to 850°C at a rate of 10°C·min⁻¹ under a flowing 5% H2/N2 mixture (20 mL/min).

Electrochemical characterisation and measurements

Cyclic voltammetric and chronoamperometric measurements were performed on a CHI 660 electrochemical workstation (CH Instruments, Austin, TX) in a standard three-electrode cell that used a platinum wire as the counter electrode and an Ag/AgCl electrode as the reference electrode. The working electrode was prepared as follows: 2.5 mg of Pt-xCeO2/Graphene catalyst was dispersed in a mixture of 0.5 mL ethanol and 7.5 μL Nafion®. After ultrasonication for 30 minutes, 5 μL of the suspension was transferred to a glass carbon (GC) electrode surface, which was polished with Al2O3 by a microsyringe. The GC electrode was then dried in air at room temperature.

The cyclic voltammogram (CV) experiments for hydrogen absorption/desorption were performed in a 1 M N2-saturated H2SO4 aqueous solution. The potential was cycled from -0.25 to 1.25 V at a scan rate of 50 mV/s. The CVs for CH3OH oxidation were performed in a mixture of N2-saturated 0.5 M H2SO4 and 0.5 M CH3OH. Here, the potential was cycled from 0 to 1 V at a scan rate of 50 mV/s. The chronoamperometric curves were recorded for 1000s at a fixed voltage of 0.7 V in the same 0.5 M H2SO4 and 0.5 M CH3OH mixed solution.

Results and Discussion

To obtain cerium oxide with different morphologies/crystal-planes, we used a simple hydrothermal method for its synthesis. The hydrothermal process obeys the so-called dissolution/recrystallisation mechanism15, 18, such that different types of cerium oxide can be obtained by adjusting the hydrothermal temperature and concentration of NaOH. As seen from the TEM and high-resolution TEM (HRTEM) images shown in Figure 1, rCeO2, cCeO2, and pCeO2 all possess uniform morphologies. In Figure 1a and 1d, the rod-shaped CeO2 has a diameter of ca. 12 nm and a length distribution between 50 and 300 nm. Figure 1d shows two interplanar spacings of 0.198 nm and 0.269 nm, which indicate the (220) and (200) planes. This configuration reflects the rod-shaped CeO2 enclosed by the (110) and (100) facets, as Figure 1g shown. From the other images in Figure 1, the cube-shaped CeO2 shows an edge length distribution between 20 nm and 50 nm, and the polyhedra-shaped CeO2 shows a diameter of ca. 10 nm. Two interplanar spacing at 0.274 nm and 0.279 nm can be observed from Figure 1e, which indicate the (200) planes of cCeO2. This configuration reflects that only the (100) facet is enclosed on the cube-shaped CeO2, as shown in Figure 1h. Finally, Figure 1f shows interplanar spacings of 0.278 nm, 0.311 nm and 0.316 nm, which indicate that the polyhedra-shaped CeO2 is enclosed by the (111) and (100) facets, as shown in Figure 1i. The further verification of the formation of cerium oxide with different morphologies/crystal-planes can be conducted with the help of XRD analysis (see Fig. S2).

Figure 1. TEM and HRTEM images of rod-shaped CeO2 (a, d), cube-shaped CeO2 (b, e), and polyhedra-shaped CeO2 (c, f).

Figure 2 shows the H2-TPR of rod-shaped CeO2, cube-shaped CeO2, and polyhedra-shaped CeO2.

Figure 2 shows the H2-TPR of rod-shaped CeO2, cube-shaped CeO2, and polyhedra-shaped CeO2. The low temperature peak (below 600°C) is caused by the reduction of the surface capping oxygens and the high temperature area (above 600°C) is caused by the reduction of bulk oxygen.20, 21 The low-temperature area of the CeO2 reduction profile indicates that this material is a better catalyst for low-temperature oxidation reactions.12 The initial surface reduction temperature represents the surface reducibility. From Figure 2, rod-shaped CeO2 have the lowest initial surface reduction temperature (approximately 200°C). Cube-shaped CeO2 and polyhedra-shaped CeO2 (approximately 275°C) have similar starting temperatures (approximately 280°C and 275°C, respectively). In the curve of rod-shaped CeO2, the low temperature reduction peak area is significantly larger than the other reduction peak areas. This can be attributed to the presence of a larger number and higher density of surface oxygen vacancies, as well as a large number of reactive oxygen species on the surface of the rod-shaped CeO2.12, 20 This result suggests that rod-shaped CeO2 have the best surface reducibility among the differently shaped CeO2 catalysts investigated.

As we have demonstrated in our H2-TPR experiment, rod-shaped CeO2 have the best surface reducibility. Mai et al.14 and Si et al.15 have investigated the oxygen storage behaviour of ceria with different crystal-planes and shapes and found that...
rod-shaped CeO₂ with surface structures dominated by (110) and (100) facets are more reactive towards CO oxidation than CeO₂ shaped differently. These groups indicated that this result could be attributed to the higher concentration of oxygen vacancies of CeO₂ on the (110) facet.

Figure 3a shows the morphology of the Pt-rCeO₂/Graphene composite. As seen, a distinct crinkled structure of the graphene can be observed. Figure 3b is a typical EDS spectrum of Pt-rCeO₂/Graphene, which corresponds to the data in Figure 3a. The data reveal that the mass fractions of Pt and rCeO₂ are 17.39% and 28.76%, respectively. Notably, these values are essentially consistent with our initial design. XRD patterns of graphene oxide (GO), rCeO₂/RGO, Pt-rCeO₂/Graphene composites were shown in Fig. S3.

To investigate the electrochemical performance of the catalysts by doping with differently shaped CeO₂, we have recorded the cyclic voltammograms of Pt-rCeO₂/G, Pt-pCeO₂/G, Pt-cCeO₂/G, and Pt/G in 0.5 M H₂SO₄ + 0.5 M CH₃OH solutions at scan rates of 50 mV/s. The corresponding data are summarised in Table 1. The catalytic performance of methanol electrooxidation can be described by the forward scanning peak current density (Iₜ) and the onset potential of the cyclic voltammograms curves. As seen in Table 1, Pt-rCeO₂/G and Pt-pCeO₂/G show relatively high forward scanning peak current densities of 286.7 mA/mg and 284.4 mA/mg, respectively. The analogous value for Pt-cCeO₂/G is only 235.6 mA/mg. We note that Pt-rCeO₂/G gives an ultra-low onset potential of 0.152 V; however, Pt-pCeO₂/G and Pt-cCeO₂/G give onset potentials of 0.186 V and 0.21 V, respectively. These results suggest that Pt-rCeO₂/G has superior catalytic activity for methanol electrooxidation relative to the other catalysts investigated.

According to the literature⁸, twenty, a high ratio of the forward anodic peak current density (Iₜ) to the backward anodic peak current density (Iₜ), Iₜ/Iₚ, implies that the catalyst has a stronger tolerance to carbonaceous species formed during the anodic electrooxidation of methanol. From Table 1, Pt-rCeO₂/G has the highest Iₜ/Iₚ ratio of 2.11 compared to the other materials, which indicates that the Pt-rCeO₂/G composite has the highest tolerance to carbonaceous species. We note that the ratios (Iₜ/Iₚ) of Pt-pCeO₂/G (1.71) and Pt-cCeO₂/G (1.73) are very similar. These results are consistent with the results from the H₂-TPR experiments that showed rCeO₂ ((110), (100)) to possess the best surface reducibility, whereas the surface reducibility of cCeO₂ ((110)) and pCeO₂ ((100), (111)) are similar. As seen in Figure 6, the Pt-rCeO₂/G and Pt-pCeO₂/G composites clearly have higher forward scanning peak current densities than the Pt-cCeO₂/G and Pt/G composites (see also Table 1). These results suggest that the rod-shaped CeO₂ enclosed by the (110), (100) facets can more effectively promote the performed of the catalyst for methanol electrooxidation than the cube-shaped CeO₂ enclosed by the (100) facet or the polyhedra-shaped CeO₂ enclosed by the (111), (100) facets. Moreover, the rod-shaped CeO₂ can greatly enhance the catalyst’s resistance to poisoning by carbonaceous species.

Table 1. Data corresponding to the results from the cyclic voltammetry experiments.

<table>
<thead>
<tr>
<th>Catalyst</th>
<th>Anodic Peak Current Density (mA/mg)</th>
<th>Onset Potential (V)</th>
</tr>
</thead>
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<tr>
<td>Pt-rCeO₂/G</td>
<td>286.7</td>
<td>0.152</td>
</tr>
<tr>
<td>Pt-pCeO₂/G</td>
<td>284.4</td>
<td>0.186</td>
</tr>
<tr>
<td>Pt-cCeO₂/G</td>
<td>235.6</td>
<td>0.21</td>
</tr>
<tr>
<td>Pt/G</td>
<td>100</td>
<td>0.7</td>
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Figure 6. Chronoamperometry curves of Pt-rCeO₂/G, Pt-pCeO₂/G, Pt-cCeO₂/G, and Pt/G in mixed solutions of 0.5 M H₂SO₄ + 0.5 M CH₃OH at 0.7 V for 1000s.
Chronoamperometric curves are usually employed to evaluate the activity and stability of a catalyst. Figure 6 shows the chronoamperometry curves of Pt-rCeO$_2$/G, Pt-pCeO$_2$/G, Pt-cCeO$_2$/G, and Pt/G in mixed solutions of 0.5 M H$_2$SO$_4$ + 0.5 M CH$_3$OH at 0.7 V for 1000 s. Because of poisoning caused by carbonaceous species such as CO$_{ads}$ and CHO$_{ads}$, all of the curves for each catalyst shown in Figure 6 display a sharp drop at the outset of the experiment. Compared with cube-shaped CeO$_2$, the activities of the catalysts doped by rod-shaped CeO$_2$ and polyhedra-shaped CeO$_2$ decrease rather gradually. After 1000 s of continual measurements, the current density of the Pt-rCeO$_2$/G catalyst retains an $i/i_0$ ratio that is approximately 3.0, 2.1, 2.0 times higher than the Pt/G, Pt-cCeO$_2$/G, and Pt-pCeO$_2$/G catalysts, respectively. These results clearly indicate that CeO$_2$ can promote a higher activity and stability to the catalyst than either cCeO$_2$ or pCeO$_2$.

Collectively, the data indicate that rod-shaped CeO$_2$ enclosed by the (110) and (100) facets can more effectively enhance the catalyst’s activity and resistance to poisoning by carbonaceous species for DMFCs than either cube-shaped CeO$_2$ enclosed by the (100) facets or polyhedra-shaped CeO$_2$ enclosed by the (111) and (100) facets. The results of calculations using simulated amorphisation and recrystallisation (A&R) suggest that the formation energies for oxygen vacancies follows the order (110)<(100)<(111), which implies that oxygen vacancies are easier to form for rCeO$_2$ enclosed by the (110) and (100) facets. Because the (100) facet is a polar surface, the concentration of oxygen vacancies for cCeO$_2$ is as low as pCeO$_2$, which is enclosed by the (111) and (100) facets. For this reason, rCeO$_2$ shows more reactivity towards CO oxidation than either pCeO$_2$ or cCeO$_2$. Further, pCeO$_2$ shows similar reactivity to cCeO$_2$, even though it has been reported that pCeO$_2$ shows higher reactivity.

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Notes and references
Scheme 1

(oxidized graphene) +

(rod-shaped CeO₂) + (cube-shaped CeO₂) + (polyhedra-shaped CeO₂)

+ + +

(Pt) (Pt) (Pt)

II II II

[Images of synthesized materials]
Figure 1
Figure 2

H₂-TPR of rod-shaped CeO₂, cube-shaped CeO₂, and polyhedra-shaped CeO₂.

Figure 3
Figure 6

![Graph showing current density vs. time for various catalysts](image-url)
Table 1. The corresponding data of the cyclic voltammograms data.

<table>
<thead>
<tr>
<th>Catalyst</th>
<th>ESA</th>
<th>Onset potential</th>
<th>$I_{\text{peak}}$</th>
<th>$I_f$</th>
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<tr>
<td>Pt-rCeO$_2$-G</td>
<td>62.6</td>
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<td>0.698</td>
<td>126.2</td>
<td>1.43</td>
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Graphical Abstract