



# One-pot synthesis of octahedral platinum nanoclusters with enhanced electrocatalytic activities



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## ABSTRACT

Octahedral platinum nanoclusters were successfully prepared by one-pot hydrothermal with the synergetic effect of the glucose and cetyl trimethyl ammonium bromide. In this study, the effects of the structure regulator and reaction time on properties of products were reported. Meanwhile the growth mechanism involved in the formation of octahedral platinum nanoclusters has been discussed. Due to its special structure, the octahedral platinum nanoclusters exhibited a better methanol oxidation activity than the commercial Pt/C catalysts, whose mass activity was 1.86 times greater than that of commercial Pt/C.

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## 1. Introduction

Because of their excellent physical and chemical properties, platinum nanoparticles have good application prospects in many fields [1–6]. Platinum nanoparticles play key roles in many industrial catalysts processes, due to their excellent catalytic activity and stability [7]. However, there are many problems, e.g., scarce reserves, high cost, harsh process conditions, which have limited the widespread use of platinum nanoparticles [8–11]. During the past several years, extensive efforts have been made to improve the catalytic activity, stability and reduce the usage amount of platinum. It is known that the catalytic property of platinum nanoparticles strongly depends on their structure, which can be enhanced by tuning their morphology, size, etc. [12–17]. Based on the size effect of nanomaterials, improving the specific surface area is one of the effective ways to perfect the mass activity and utilization efficiency. When the catalyst quality is equal, the mass activity of the catalyst is larger whose size is smaller and specific surface area is larger [18]. Lim et al. have synthesized Pd–Pt bimetallic nanodendrites consisting of a dense array of Pt branches on a Pd core by reducing  $K_2PtCl_4$  with L-ascorbic acid, and the Pd–Pt

nanodendrites exhibited 2.5 times more active on the basis of equivalent Pt mass for the ORR than the state-of-the-art Pt/C catalyst [19]. Seleem et al. synthesized ultrathin Pt–Cu alloy nanosheets of 4–6 atom thickness with tunable lateral size from 10 to 50 nm, their mass activity is 2.7 times than that of commercial Pt/C [20]. Although the platinum nanoparticles have been synthesized with various methods, but a simple, rapid and cost-effective preparation method still remains a challenge.

Herein, a brief and rapid hydrothermal method was proposed to directly produce octahedral platinum nanoclusters (OTPtNCs) with the synergetic effect of the glucose and cetyl trimethyl ammonium bromide (CTAB). It was carried out without the need for any template and seed-mediated growth. We have revealed the growth mechanism and identified the key factors that influence the formation of the OTPtNCs by spatial control. Meanwhile the electric catalytic properties of the OTPtNCs were tested.

## 2. Experimental

### 2.1. Synthesis of octahedral platinum nanoclusters

In a typical synthesis, 0.1 mL of  $H_2PtCl_6$  solution (0.2 M), 5 mL of CTAB solution (50 mM) and 40 mL of glucose solution (0.5 M) were added into the Teflon liner, respectively. After stirring for 10 min, the mixed solution was transferred into a 100 mL Teflon-lined autoclave. The autoclave was then maintained at 180 °C for 1 h before it was cooled down to room temperature. The products

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were separated via centrifugation and further purified by ethanol three times. Finally, the products were redispersion into ethanol. Also, the effects of the amount of glucose and CTAB on the product were studied.

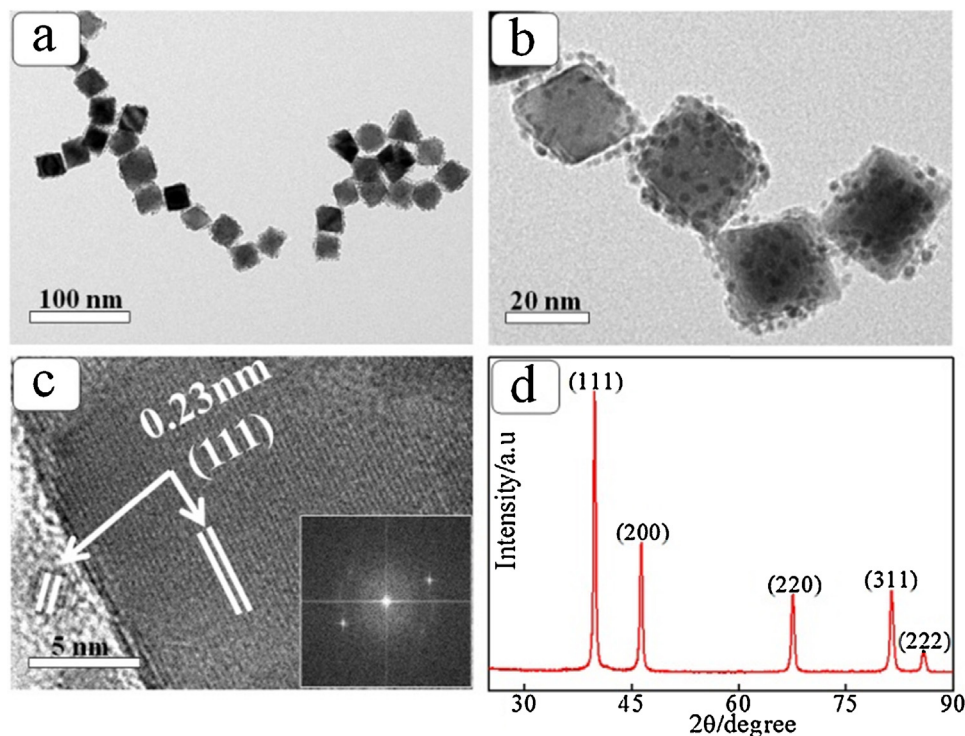
## 2.2. Electrochemical measurements

Electrochemical experiments were performed with a CHI 660E electrochemical workstation (ChenHua, Shanghai). Cyclic voltammetry (CV) was carried out by using a standard three electrode cell system with the glassy carbon electrode (GCE,  $d = 3$  mm) as the working electrode, a Pt foil as the counter electrode, and a saturated calomel electrode (SCE) as the reference electrode. Prior to the use, the GCE was polished carefully with  $0.05 \mu\text{m}$  alumina powder for 1–2 min and rinsed with deionized water, followed by sonication in ethanol and doubly distilled water, successively. Then, the electrode was allowed to dry in the air. The catalyst dispersion was prepared by mixing catalyst in ethanol. For cyclic voltammogram,  $2 \times 10^{-3}$  mL of the catalyst suspension and  $2 \times 10^{-3}$  mL of 0.05 wt% Nafion solution were dropped on the surface of GCE, respectively. The Pt loading of each sample was  $0.02 \text{ mg cm}^{-2}$ . The electrodes were immersed in 0.5 M  $\text{HClO}_4$  solution, and the potential was scanned from  $-0.2$  to  $1.2$  V at a scan rate of  $50 \text{ mV/s}$  to obtain the cyclic obtain the CVs. The electrochemical active surface areas (ECSA) of the catalysts were estimated by the region for hydrogen desorption. Methanol oxidation (MOR) activities of the catalysts were evaluated in a solution containing 0.5 M  $\text{HClO}_4$  and 0.5 M MeOH with a scan rate of  $50 \text{ mV/s}$ . CA curves were recorded at  $0.6$  V in a solution containing 0.5 M  $\text{HClO}_4$  and 0.5 M MeOH for 3600 s to demonstrate the stability of the various catalysts.

## 3. Results and discussion

The products mainly contained the OTPtNCs (Fig. 1a and b and Fig. 2). The TEM images in Fig. 1a and b show the OTPtNCs at different magnifications. An average particle size was measured to be  $24.3 \text{ nm}$ , and the surfaces of the OTPtNCs were covered by small platinum nanoparticles uniformly. Fig. 1c shows a high-resolution TEM (HRTEM) image of a single OTPtNCs. As shown in Fig. 1c, the small platinum nanoparticles have average diameter of  $2.5 \text{ nm}$ . The fringes with lattice spacing of  $0.23 \text{ nm}$  can be indexed as (111) plane. Both the HRTEM image and the corresponding Fourier transform (FT) pattern indicated that the OTPtNCs were single-crystalline. It can be observed that there was a thin LaMer model [21] carbon layer on the OTPtNCs surface (Fig. 1c and Fig. 3). The LaMer model carbon layer anchored the Pt small particles, and enhanced the dispersion of the products. The powder X-ray diffraction (PXRD) pattern of the products (Fig. 1d) indicates that the OTPtNCs have a face-centered cubic (fcc) phase. The reflections at  $39.8^\circ$ ,  $46.3^\circ$ ,  $67.4^\circ$ ,  $81.5^\circ$ , and  $85.9^\circ$   $2\theta$  values corresponded to the (111), (200), (220), (311), and (222) planes of the fcc Pt phase, respectively.

To understand the growth process of the OTPtNCs, the intermediate products obtained by varying the reaction time during the hydrothermal process. As shown in Fig. 4a, small irregular nanoparticles with an average size of  $10 \text{ nm}$  or less were obtained, when the reaction time was  $20 \text{ min}$ . When the reaction time was increased to  $40 \text{ min}$ , it was worth pointing out that the morphology of platinum nanocrystals gradually structured, but there were still no small Pt nanoparticles in the system. It was seen that the OTPtNCs were formed after reaction for  $60 \text{ min}$ . When the reaction time was further increased to  $90 \text{ min}$ , we found that the OTPtNCs disappeared and converted into multipetal Pt nanocrystals with average diameter of  $34 \text{ nm}$ . Time sequential evolution



**Fig. 1.** a) Low-magnification TEM; b) high-magnification TEM; inset shows the geometrical model; c) HRTEM images of the single; d) XRD pattern of products; inset shows the FFT pattern of the single;  $t = 60 \text{ min}$ .

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