



## Featured Letter

## High efficiency nitrogen-doped core-shell carbon spheres as counter electrodes for dye-sensitized solar cells

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## ABSTRACT

A novel metal-free conductive catalyst is designed based on N-doped core-shell carbon spheres (N-CCS) with rich active sites. Furthermore, the doping of N atoms activated the fully interconnected mesoporous basal planes of carbon spheres. Corresponding electrochemistry (Cyclic voltammetry, electrochemical impedance spectroscopy, and Tafel polarization measurements) test discovered the excellent electrocatalytic activities in the  $I^-/I_3^-$  redox reaction of dye-sensitized solar cells (DSSCs), suggesting to result from the improvement of the surface electronic properties of CCS. DSSCs with N-CCS as the counter electrode (CE) achieve power conversion efficiency (PCE) of 7.89%, higher than to Pt's value (7.48%) in the same test environment, demonstrating as a hopeful catalyst for low-cost DSSCs.

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## 1. Introduction

DSSC as a promising solar cell with high photoelectric conversion efficiency has been favored by researchers. The counter electrode (CE) inside plays an important role in collecting electrons and catalyzing the reduction of  $I^-/I_3^-$  in the whole photoelectric conversion process for DSSC [1]. Pt is widely used as CE due to its good conductivity and catalytic capacity. However, it is a pity that Pt is a noble and scarce metal [2]. Now researchers have been developing other CE materials for the replacement of Pt, such as conducting polymers [3], and inorganic compounds [4]. But some of these materials show either low PCE or the synthesis of them is complicated. It is very desirable to find novel materials that are low-cost, and should achieve good electrochemical performance as well. The nanostructured CE materials can match these points, i.e., possessing fast electronic transport, high specific surface area, large pore volume, low cost, and easy to synthesis.

M Yu et al. have developed N-doped reduced graphene oxide, which is then used as CE and reaches a PCE of 5.56% [1]. However, the fabrication processing is very complicated and time-consuming. Here, we put forward a reasonable design in which highly uniform N-CCS with a size of around 300 nm are controllably fabricated by using a low-cost resorcinol as a carbon source. The cell using N-CCS as CE shows a PCE of 7.89% that is higher than

that of Pt (7.48%), demonstrating that N-CCS is of great potential as alternatives for Pt. The present work affords a very simple and efficient method to constitute resourceful carbon-based CEs for Pt replacement in DSSCs.

## 2. Experimental

The synthesis of CCS and N-CCS are shown in [Supporting Information](#).

## 2.1. Characterization and measurements

Raman spectroscopy by a Rigaku/Max-3A X-ray diffractometer with Cu K $\alpha$  radiation ( $\lambda = 1.54178 \text{ \AA}$ ). X-ray photoelectron spectroscopy was conducted using a Smart Lab 9 KW to study the chemical states. The surface morphology of CEs was observed via a field-emission scanning electron microscope (JSM-6701F, JEOL accelerating voltage of 5 kV) and transmission electron microscopy (TEM, JEOL-2010, operating voltage of 200 kV), high-resolution TEM (HRTEM, JEOL-2010). Cyclic voltammetry (CV) curves were performed in a supporting electrolyte consisting of 10 mM LiI, 1 mM  $I_2$ , and 100 mM LiClO<sub>4</sub> in acetonitrile and carried out in a three-electrode system. Pt thin film was used as the CE and Ag/AgCl with saturated KCl solution as reference electrode. Electrochemical impedance spectroscopy (EIS) was recorded in a frequency range of 0.1–10<sup>6</sup> Hz and at alternating current voltage amplitude of 10 mV. Tafel polarization kept quasi stationary conditions with the voltage range –1 to 1 V at a scan rate of 50 mV/s.

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Photovoltaic measurements of photocurrent density-voltage ( $J$ - $V$ ) curves were measured by a solar light simulator under an illumination of  $100 \text{ mW cm}^{-2}$ , AM 1.5 and using a digital source meter. All the measurements were performed at room temperature.

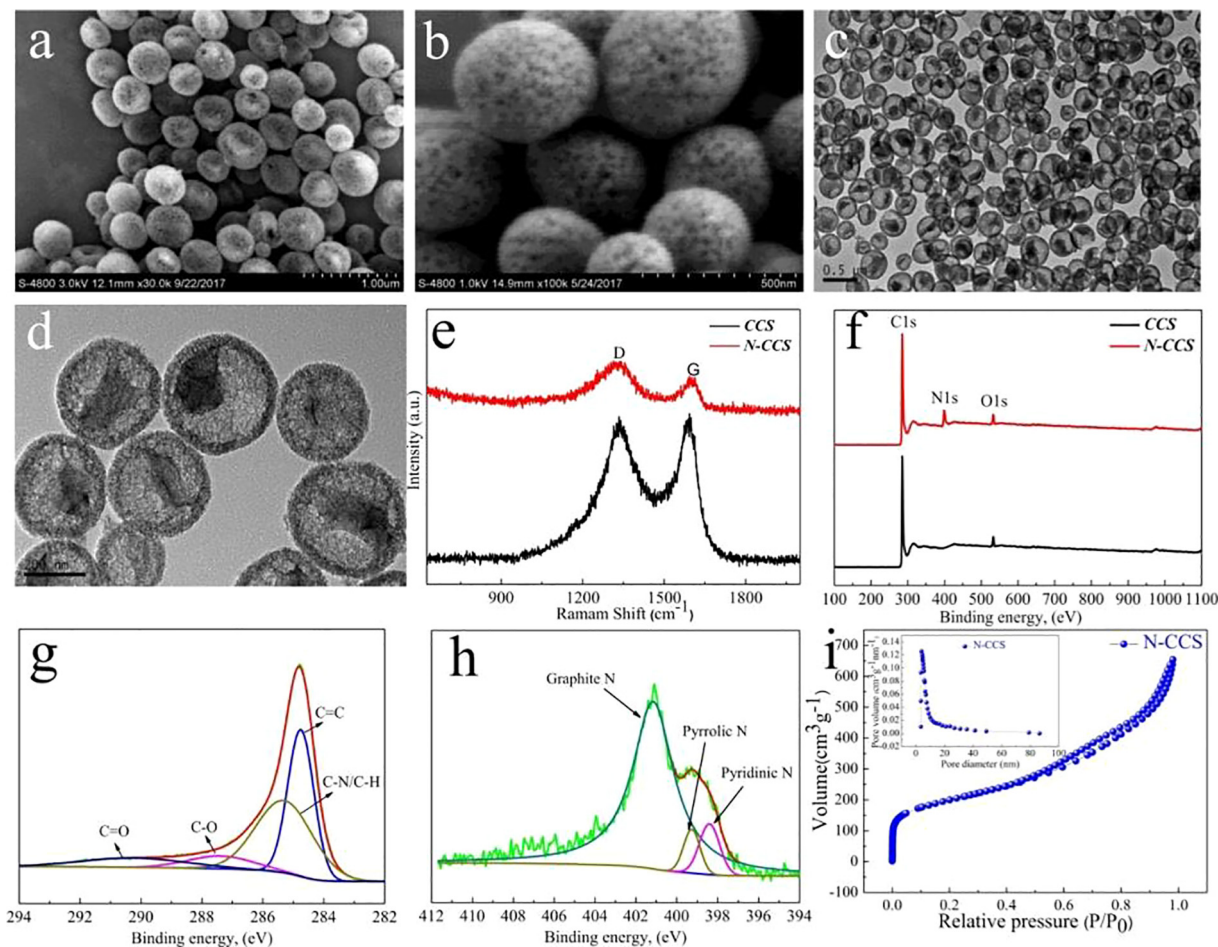
### 3. Results and discussion

Fig. 1(a–d) demonstrates the morphology and structure of as prepared N-CCS. N-CCS have two characteristics, the one is the uniform mesoporous shapes, the size of mesoporous are about 10 nm which is presented in the SEM images Fig. 1a and b. The second one is core-shell structure, the size of N-CCS is about 300 nm, the internal structure of N-CCS can be clearly displayed in TEM images Fig. 1c and d. Composition analysis based on both Raman spectroscopy and X-ray photoelectron spectroscopy, in Fig. 1e, for the pristine CCS and N-CCS have different D peaks and G peaks. It is also noted that the  $I_D/I_G$  values of N-CCS is greater than that of CCS, and show more defects within the as-made sample matrix after doping with nitrogen, which enhance the electrocatalytic activity of CCS [5]. The chemical composition is further examined by XPS. As shown in Fig. 1f, the typical signals of carbon, oxygen, and nitrogen elements are found. Fig. 1g establishes a relatively main C=C peak at 284.4 eV, C–N/C–H (285.2 eV), C–O (286.6 eV), and C=O (287.5 eV) configurations. In Fig. 1h, it can be clearly observed that three typical N species are recognized by the bonding states of N atoms, including pyridinic N (398.4 eV), pyrrolic N (399.3 eV), and graphite N (401.1 eV). The high-content graphite N species could modify the electronic structure of N-CCS basal

planes and provide extra defects, thus assisting the reduction of  $I_3^-$ , which will be further photoelectric performance. For another, the structure of the carbon ball also gives a higher degree of performance improvement, the particular core-shell structure may lead to a large specific surface area, which is further demonstrated by the BET test shown in Fig. 1i, and the BET test of N-CCS achieves  $690 \text{ m}^2 \text{ g}^{-1}$ , the larger surface area can absorb more electrons. Meanwhile, the BJH method was used to analyze the pore size distributions of N-CCS, which is inserted in Fig. 1i, the pore size distribution with about 7.8 nm. The interconnected mesoporous networks can help many electrolyte ion diffusion paths along different directions with a short distance, which can shorten the reaction time effectively.

To evaluate the catalytic activity of N-CCS, cyclic voltammetry (CV) measurements were carried out with a three-electrode system [6]. In Fig. 2a,  $I_3^-$  decomposes to one  $I^-$  and  $I_2$ , followed by the reduction of the I atoms after the dissociation of  $I_3^-$  on the CE surface. Two typical pairs of oxidation and reduction peaks in the CV curves were clearly observed and shown in Fig. 2b, the catalytic behaviors is evaluated by two important parameters of the peak-to-peak separation ( $E_{pp}$ ) and peak current density. The gap between the Ox1 peak and the Red1 peak, which is defined as  $E_{pp}$ . The smaller gap of  $E_{pp}$  mirrors the higher catalytic activities for CEs [7]. The  $E_{pp}$  of N-CCS is 0.352 V and smaller to that of Pt's value (0.365 V). Meanwhile, the higher peak intensity means stronger current density. Therefore, the catalytic behavior of N-CCS is than that of Pt.

Electrochemical impedance spectra were recorded in a symmetrical cell (electrode/electrolyte/electrode) to assess



**Fig. 1.** (a), (b) SEM images of N-CCS. (c), (d) TEM images of N-CCS. (e) Raman spectra of CCS and N-CCS. (f) XPS spectra of CCS and N-CCS. (g) C1s spectra. (h) N1s spectra. (i) Nitrogen adsorption and desorption isotherms and pore size distributions of N-CCS.

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