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## Fabrication of Pt-doped carbon aerogels for hydrogen storage by radiation method

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### ABSTRACT

A radiation method was investigated to fabricate Pt-doped carbon aerogels (CAs–Pt). The physicochemical properties of the pristine CAs and CAs–Pt were systematically characterized by X-ray diffraction, scanning electron microscope, transmission electron microscopy, and nitrogen adsorption measurements. The results showed that not only a great number of Pt nanospheres but also many Pt nanoparticles presented in the network of CAs after radiation. The influence of Pt doping on the hydrogen uptake capacity of CAs was studied. In comparison with the pristine CAs, it was remarkable that the hydrogen uptake capacity of the CAs–Pt had been significantly enhanced, which was contributed by the hydrogen spillover of Pt.

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### Introduction

The global warming due to the slather of traditional energy resources has attracted more and more attention [1,2]. So far the fossil fuels still are main energy resources with the increase of global energy demand, which brings us the increasingly severe environmental and social problems [3,4]. To solve the problems of global warming, it is important to develop renewable energy sources instead of the fossil fuels [5,6]. Hydrogen is regarded as one of the most promising alternative energy carrier for both transport and mobile applications, due to its large energy density, slow mass density and nonpolluting nature [7–9]. However, the storage of hydrogen is the major challenge for realization of hydrogen

technology in vehicular applications [10]. Since hydrogen is gaseous at ambient temperature and pressure, the compression of hydrogen consumes lots of energy and is inefficiency. Generally, the methods of hydrogen storage include liquidized hydrogen, adsorption in metal alloys, adsorption in liquid organic metal hydrides or adsorption in porous materials. Nevertheless, none of these storage methods could reach the requirements of onboard vehicular applications until now [11–15]. In order to meet the demands of vehicular applications, the US Department of Energy (DOE) has set the system capacity targets of 7.5 wt% with the condition of ambient temperature and a maximum pressure of 12 bar [16]. Hence, it is clear that the technological bottleneck of hydrogen applications is development of efficient and reliable hydrogen storage system with high capacities for mobile applications.

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Among various methods to hydrogen storage, adsorption based hydrogen storage on porous materials received considerable interest due to their faster adsorption-desorption kinetics, high surface areas, excellent cyclability, light weight and chemical inertness [17]. A large number of porous materials such as zeolites, porous polymer, metal organic frameworks and porous carbons have been extensively investigated as potential hydrogen storage media [18–20]. Carbon aerogels are the most widely investigated sorbents for reversible hydrogen storage because of their high surface area, low density, high chemical and thermal stability, and they are easy to large scale preparation with low cost [21]. Nevertheless, the low binding energy of hydrogen ( $6 \text{ kJ mol}^{-1}$ ) requires that the cryogenic temperature (77 K) is utilized for storage of hydrogen in these materials [22]. Experimental findings showed that the maximum storage capacities of pristine carbon aerogels at room temperature was below 1 wt %, even with specific surface areas up to  $2000 \text{ m}^2 \text{ g}^{-1}$  and highly developed pore structures [23–25]. For instance, Tian et al. reported the hydrogen storage value of pristine carbon aerogels with the specific surface area of  $2206 \text{ m}^2 \text{ g}^{-1}$  were 4.8 wt% at 77 K and 0.5 wt% at room temperature under the same pressure (45 bar) [26]. To enhance the hydrogen uptake capacity of carbon aerogels at room temperature, one of the effective methods is to dope carbon aerogels with the noble metal, such as platinum, via the spillover effect [27–33]. Various methods have been presented to incorporate metal onto the sorbents, like the wet impregnation, atomic layer deposition (ALD), chemical vapor deposition (CVD) and plasma-assisted doping [25,34–36]. Not only the distribution and size of the metal particles but also the interaction between the supports and metal particles depend on the doping method. Thus, the doping method influences the hydrogen uptake capacity of metal-doped sorbents.

In this paper, we have reported a novel method (radiation method) to fabricate Pt-doped carbon aerogels. The main advantage of radiation method is its versatility. For instance, it is well known that radiation process allows formation of nonequilibrium metal clusters such as Au cores surrounded by the Ag shell [37]. These structures are not achieved by traditional impregnation method, where the formation of cluster is obtained by high-temperature treatment, which generates equilibrium structures. The products were characterized by X-ray diffraction (XRD). The inductively couple plasma atomic emission spectrometer (ICP-AES) was used to analyze the content of Pt. The microstructure of Pt-doped carbon aerogels was determined using the transmission electron microscopy (TEM) and scanning electron microscope (SEM). Impact of Pt doping on the hydrogen uptake capacities of carbon aerogels had been studied in details.

## Experimental section

### Preparation of carbon aerogels

Carbon aerogels (CAs) were synthesized via the established procedures as our previous work [38]. As the first step, the RF hydrogels were fabricated through a sol-gel polymerization process of resorcinol (R) and formaldehyde (F). A mount of

resorcinol, formaldehyde, sodium carbonate, and deionized water were mixed in a glass vial and holding the mixture at 333 K for 6 days. The molar ratio of R/F was fixed at 1/2 and sodium carbonate was used as the catalyst. Next, crosslinked RF gels were exchanged with acetone and dried from supercritical  $\text{CO}_2$ . In the end, RF aerogels were disposed to pyrolysis in a tubular oven at 1323 K under argon atmosphere to obtain the CAs.

### Preparation of Pt-doped carbon aerogels

The preparation procedure of Pt-doped CAs is shown in Fig. 1. A certain amount of chloroplatinic acid (1 g) was added to 20 ml of deionized water, which was stirred for 30 min in a glass vial at ambient temperature, forming a  $\text{PtCl}_2$  solution. Next, 0.2 g of CAs monoliths were immersed in 10 ml of  $\text{PtCl}_2$  solution. In order to scavenge the oxidizing radicals generated during irradiation, the proper amount of 2-Propanol was added. The mixture was deaerated by bubbling with nitrogen for 15 min to remove oxygen solvated in it and subsequently ultrasonicated for 1 h to well disperse Pt ions into CAs. Then the mixture was sealed in the reaction vial and irradiated for a total absorbed dose of 500 kGy in the  $^{60}\text{Co}$   $\gamma$ -ray radiation source at ambient temperature. The CAs after irradiation were washed with abundant ethanol and deionized water. Finally, the Pt-doped CAs were derived from drying of the resultant monoliths at 333 K in a vacuum drying oven for 5 h. The Pt-doped CAs were labeled as CAs–Pt in this paper. The Pt content of CAs–Pt is 0.21 wt%, which was measured by the ICP-AES.

### Characterization

The X-ray diffraction measurements were carried out on a Panalytical X'Pert Pro X-ray diffractometry with nickel-filtered  $\text{Cu K}\alpha$  radiation as the X-ray source. X-ray photoelectron spectroscopy (XPS) was used to characterize the chemical states of the constituent elements. XPS spectra were acquired in XSAM 800 system and decomposed into Gaussian components by a least-square fitting method. The morphologies and energy dispersive spectroscopy (EDS) analysis of the samples were characterized by transmission electron microscope (TEM, JEM-200CM, 200 kV) and scanning electron microscope (SEM, Nova 600i). The nitrogen adsorption-desorption isotherms of the samples were obtained by using a Quantachrome Autosorb-1 instrument. The specific surface areas were calculated with the Brunauer-Emmett-Teller (BET) method, and pore size distributions with the Density Functional Theory (DFT) model [39,40]. Hydrogen sorption properties of the samples were carried out at 298 K and up to pressure of 50 bar by using a Sievert' apparatus. The pressure-composition relationships were obtained by calculating the hydrogen storage capacity in wt%.

## Results and discussion

The powder X-ray diffraction results of the CAs–Pt and pristine CAs samples are presented in Fig. 2. For the pristine CAs, the two broad diffraction peaks at  $23^\circ$  and  $43^\circ$  are observed, which correspond to the (002) and (100) plane reflections of

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