



Hierarchical porous carbon derived from rice husk as a low-cost counter electrode of dye-sensitized solar cells



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ABSTRACT

The porous carbon with a high surface area is prepared from rice husk through pyrolysis and chemical activation and explored as the counter electrode of dye-sensitized solar cells. Structure and texture analysis reveals that the as-prepared porous carbon has a hierarchical porous structure containing large-size mesopores and abundant micropores. Electrochemical studies indicate that the obtained hierarchical porous carbon electrode has high electrocatalytic activity for I^-/I_3^- redox reaction and enhanced electrolyte diffusion capability. These enhanced electrochemical properties are beneficial for improving the photovoltaic performance of dye-sensitized solar cells. Under irradiation of 100 mW cm^{-2} , the dye-sensitized solar cell with this porous carbon counter electrode shows a conversion efficiency of 6.32%, which is closed to that of the cell with Pt electrode (6.69%). These promising results highlight the potential application of hierarchical porous carbon derived from rice husk in more cost-effective dye-sensitized solar cells.

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1. Introduction

Dye-sensitized solar cells (DSCs) have attracted considerable attention as one of the next generation solar cells due to their low cost, simple fabrication process and relatively high conversion efficiency [1–3]. A typical DSC consists of a dye-sensitized nanocrystalline TiO_2 electrode, a catalytic counter electrode and an electrolyte containing I^-/I_3^- redox couple. The counter electrode is an indispensable component in a DSC, which serves to transfer electrons arriving from the external circuit back to the redox electrolyte and carry the photocurrent over the width of solar cell [4]. Therefore, the excellent electrocatalytic activity, high surface area, and sufficient electrical conductivity are the essential properties for a good counter electrode. Usually, a thin layer of Pt coated on a conductive substrate is used as a counter electrode in DSCs due to its high electric conductivity and superior electrocatalytic activity [5,6]. However, the high cost, limited reserve and risk of Pt corrosion by the redox species in the electrolyte would prevent it from use in large solar conversion system. Therefore, it is of high importance to develop low-cost, more corrosion stable and Pt-free counter electrode with a high electrocatalytic activity for future development of DSCs.

In recent years, some cost-effective alternatives to Pt have been extensively investigated in DSCs, such as carbonaceous materials [7–17], conductive polymer [18–21] and inorganic materials [22,23]. Clearly, all the potential alternatives must have both the high electrocatalytic activity for I^-/I_3^- redox reaction and the good electrical conductivity. Carbonaceous materials are quite attractive to replace Pt due to their low cost, high electrical conductivity, good electrocatalytic activity and chemical stability. A variety of carbonaceous materials, such as carbon black [7,8], carbon nanotubes [9,10], active carbon [11], graphene [12,13] and porous carbon [14–17], have been used as the counter electrode and exhibited reasonable performance. Among them, porous carbons hold considerable appeal for counter electrodes in DSCs because of their high surface area, well-tailored pore size, large pore volume with an interconnected pore structure, and good electrocatalytic activity. Currently, porous carbons are mainly prepared through a templating method using mesoporous silica and zeolites as hard templates, and amphiphilic block copolymers as soft templates [24,25]. However, such templating approaches usually require expensive and non-renewable raw materials and tedious preparation procedures, which limited their practical applications.

Recently, as a renewable source, biomasses have attracted much attention for their application in the preparation of porous carbon. Rice husk is a kind of agricultural biomass waste and has the advantages of being inexpensive and widely available. Activating the rice husk in the inert atmosphere produces a highly porous carbon powder with a very high surface area [26]. During recent years,

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porous carbons prepared from rice husk have attracted a great deal of attention in the field of environment protection and energy storage [27–29]. In this work, we report that the hierarchical porous carbon derived from rice husk can be used as the low-cost alternative to Pt in the counter electrode of DSCs. The hierarchical porous framework provides the pathway to ensure easy accessibility of electrolyte and fast transportation of the redox couple. Electrochemical measurements indicated that the obtained carbon electrode had high electrocatalytic activity for I^-/I_3^- redox reaction. The DSC with hierarchical porous carbon electrode had similar photovoltaic performance as that of the cell with Pt electrode in terms of short-circuit current density, open-circuit voltage and conversion efficiency.

2. Experimental

2.1. Preparation of porous carbons and carbon electrodes

The rice husk was washed thoroughly with distilled water and then dried in air at 100 °C for 24 h. The dried rice husk was carbonized at 500 °C for 3 h under nitrogen atmosphere. The resulting material is labeled as precarbonized carbon (C-1). The precarbonized product was ground, immersed in diluted HF (25%, w/w), rinsed with distilled water and dried at 100 °C for 12 h. The final product is denoted as C-2. 30 g C-2 was soaked in a KOH solution (the mass ratio of KOH to C-2 is 4:1) for 3 h. Then, the above mixture was stirred at 100 °C to evaporate water. When the mixture turned to a muddy mass, it was transferred into a tube furnace for activation under N_2 atmosphere at 800 °C for 2 h. The obtained product was washed with distilled water until the pH value of the system reached about 7, and dried at 100 °C. The dried sample was named as C-3.

150 mg obtained carbon sample was ground in a mortar with 7 ml *n*-butanol and 0.2 ml tetrabutyl titanate to obtain the carbon paste. The carbon paste was coated on fluorine-doped tin oxide (FTO) glass by doctor-blade method, and then sintered at 400 °C for 15 min to obtain carbon electrodes.

2.2. Fabrication of DSCs

The TiO_2 colloidal paste was prepared according to the procedures reported previously [30]. The TiO_2 nanocrystalline electrodes were prepared by depositing TiO_2 colloidal paste onto FTO glass and then sintered at 450 °C for 30 min in air. The obtained TiO_2 electrodes were dipped into the ethanol solution containing 0.5 mM N_3 dye for 12 h at room temperature. Afterwards, the dye-sensitized TiO_2 electrodes were rinsed with ethanol and dried in the air. A DSC was fabricated by clamping a dye-sensitized TiO_2 electrode and a counter electrode. The interelectrode space was filled with the electrolyte consisting of 0.4 M LiI, 0.05 M I_2 , 0.4 M 1-methyl-3-hexylimidazolium iodide, and 0.5 M 4-tert-butylpyridine in 3-methoxypropionitrile. The effective area of the cell is 0.2 cm^2 .

2.3. Characterization

Micromeritics ASAP 2020 instrument was used to characterize the surface area and pore structure of the carbon samples. X-Ray diffraction pattern was obtained using a Bruker D8 Advance X-ray diffractometer with Cu K_{α} radiation. Transmission electron microscopy (TEM) images were obtained on an H800 (HITACHI, Japan) electron microscope. The morphology of the carbon electrode was investigated by scanning electron microscopy (SEM, Sirion 200). Cyclic voltammetry (CV) was carried out in a three-electrode system using a CHI660C potentiostat with a Pt-foiled counter electrode and a saturated calomel reference electrode dipped in an acetonitrile solution of 0.01 M LiI, 0.001 M I_2 and 0.1 M $LiClO_4$. Electrochemical impedance spectroscopy (EIS) measurements were performed on a Solartron 1255B frequency response analyzer equipped with a Solartron 1287 electrochemical interface system. The photocurrent–voltage characteristics of DSCs were measured on Keithley 2400 Source Meter under irradiation of 450 W Xe lamp equipped with an AM 1.5G filter at the light intensity of 100 $mW\ cm^{-2}$.

3. Results and discussion

The composition of rice husk is complicated, so the process of preparing porous carbon consists of precarbonization, leaching and activation processes. The pyrolysis of the organic components in rice husk takes place at precarbonized step. When the leaching step performed, inorganic components are removed. At the activation process, the pore-opening and pore-widening effects occur simultaneously. The N_2 adsorption–desorption isotherm of C-1 sample (Fig. S1 see ESM) is of type I as the adsorption and desorption branches remain nearly horizontal, which is characteristic behavior of microporous materials. However, the isotherms of C-2 and C-3 samples (Fig. S1 see ESM) can be classified into type IV and show obvious capillary condensation step (hysteresis loop), indicating the presence of mesopores. Surface area and pore structure properties of the all studied carbon samples are summarized in Table 1. The data listed in Table 1 suggest that the pyrolysis process mainly results in the formation of micropores. The pore volume of C-1 sample is very small (only 0.15 $cm^3\ g^{-1}$). After leaching process, inorganic components (mainly silica) are removed, the BET surface area and pore volume of C-2 increase to 321.2 $m^2\ g^{-1}$ and 0.31 $cm^3\ g^{-1}$, respectively. Compared the textural characteristics between C-1 and C-2, it is observed that C-2 shows much higher mesopore surface area and volume than C-1, whilst comparable micropore surface area and volume to C-1. These results suggest that removing inorganic components from C-1 sample mainly generates mesopore structures. Activation of C-2 increases the surface area and pore volume. After activation process, the BET surface area, micropore surface area and volume, and total pore volume of C-3 sample increase remarkably from 321.2 to 1094.6 $m^2\ g^{-1}$, 231.3 to 962.4 $m^2\ g^{-1}$, 0.11 to 0.35 $cm^3\ g^{-1}$, and 0.31 to 0.61 $cm^3\ g^{-1}$, respectively. The mesopore surface area and volume of C-3 also rise from 89.9 to 132.2 $m^2\ g^{-1}$ and 0.20 to

Table 1
Surface area and pore structure characteristics of carbon samples obtained from rice husk.

Sample	S_{BET} ($m^2\ g^{-1}$)	S_{micro} ($m^2\ g^{-1}$)	S_{meso} ($m^2\ g^{-1}$)	V_T ($cm^3\ g^{-1}$)	V_{micro} ($cm^3\ g^{-1}$)	V_{meso} ($cm^3\ g^{-1}$)	V_{meso}/V_T (%)	D_p (nm)
C-1	268.7	247.1	21.6	0.15	0.13	0.02	13.3	1.9
C-2	321.2	231.3	89.9	0.31	0.11	0.20	64.5	6.7
C-3	1094.6	962.4	132.2	0.61	0.35	0.26	42.6	2.5

S_{BET} : Brunauer–Emmett–Teller surface area. S_{micro} : micropore surface area, derived from t-plot method. S_{meso} : mesopore surface area, calculated by subtraction of micropore surface area from BET surface area. V_T : total pore volume. V_{micro} : micropore volume, derived from t-plot method. V_{meso} : mesopore volume, calculated by subtraction of micropore volume from total pore volume. D_p : average pore diameter, calculated as 4 V/A by BET.

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