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Structure Characterization and Performance Test of Carbon Aerogel

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Carbon aerogels have been prepared using calvital (C) as catalyst derived *via* a sol-gel process with resorcinol (R) and formaldehyde. It was dried at open air with low surface tension. Further pyrolysis of these materials produced carbon aerogels. Carbon aerogels was investigated by scanning electron microscopy and nitrogen adsorption measurements. Electrochemical impedance and cyclic valtammogram were used to study the electrochemical permance. The BET surface areas of the carbon aerogels are from 800 m^2/g to 1000 m^2/g. Electrochemical tests show that carbon aerogel used in the electrochemical systems expresed very good capacitive.

Key Words: Carbon aerogels, Calvital, Electrochemical performance.

INTRODUCTION

Carbon aerogels are novel mesoporous materials with many properties, such as highly usable surface area, controllable pore structure and high electrical conductivity[1-3]. Carbon aerogels are usually dried supercritically with respect to carbon dioxide[4,5], it can be also dried subcritically *via* direct evaporation of the pore liquid at ambient pressure. Because of their unsure structure characteristics, carbon aerogels are widely used. Carbon aerogels belong to disordered carbon materials, they can provide maximum specific enery, long cycle life and sufficient special power, so they can be used on the negative side of the battteries[6-8]. Carbon aerogels are used as electrode materials for electric double-layer capacitors[9,10].

In this paper, the effect of nR/nC (molar ratio of resorcinol to catalyst) on the pore size distribution, surface aera, specific capacitance of the carbon aerogels was presented, along with results obtained from SEM, N_2 adsorption and electrochemical analysis including impedance spectroscopy and cyclic voltammetry.

EXPERIMENTAL

Micromeritics ASAP2420 (Micromeritics Instrument (Shanghai) Ltd.), Scanning electron microscopy(S-570), Electronic Scale (BSA423S-CW), Electrochemical Workstation (Chenhua Instrument, Inc., Shanghai, China).

Calvital, formaldehyde and acetone were purchased from Huitong Chemical Industry. Resorcinol was purchased from Jindi chemical industry. All of the materials were analytical grade.

Method: The surface microstructure of carbon aerogels were researched by scanning electron microscopy (S-570). Electrochemical measurements were carried out using CHI660B electrochemical workstation (Chenhua Instrument, Inc., Shanghai, China). Impedance spectra were recorded at open corrosion potential in the frequency range 0.05 Hz to 100 kHz.

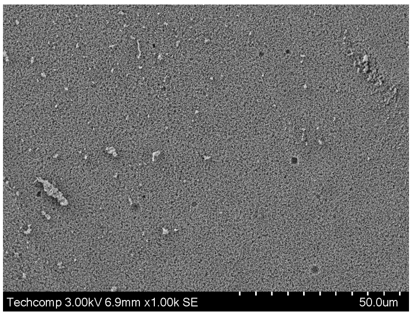
Preparation of carbon aerogel: Organic gels were prepared *via* a sol-process by resorcinol (R) with formaldehyde (F), using calvital as a base catalyst. The nR/nC ratio primarily determines the grain size. With the parameter the structure of RF gels can be controlled over a wide range[11,12].

Preparation of electrodes: The glassy carbon electrode of the exposed surface was performed by polishing mechanically to smooth surface finish, using 1.0, 0.5, 0.03 μm α-Al_2O_3 on lint. Carbon aerogel was grinded to power, mixed with 0.1 % nafion solution and then daubed that on the glassy carbon electrode. The electrodes dried in the air, which was the working electrode using in the electrochemical measurements.

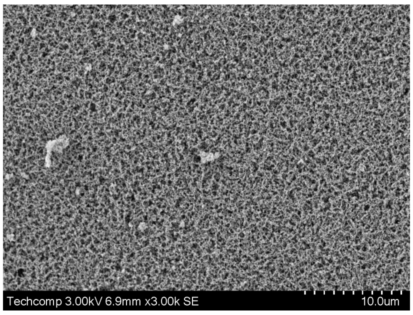
RESULTS AND DISCUSSION

Structure of carbon aerogel: SEM morphologies of carbon aerogels with different nR/nC are shown in Fig. 1(a-e). From a to e, the nR/nC of carbon aerogel were prepared at 500, 800, 1000, 1200, 1500, but the RF mass concentration was at 25 %.

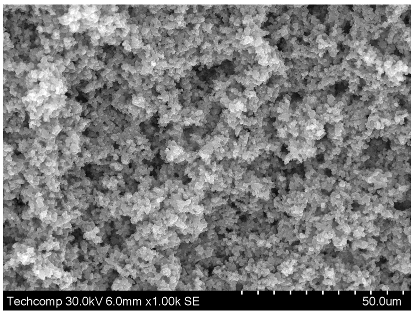
Carbon aerogel were composed of clusters of particles around several tens of nm in diameter. As seen in Fig. 1, it was



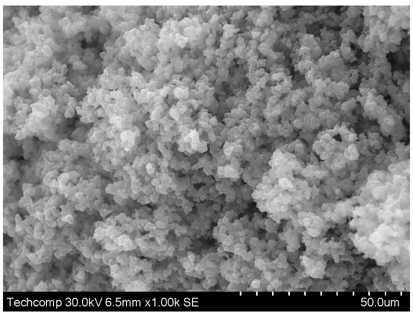
(a)



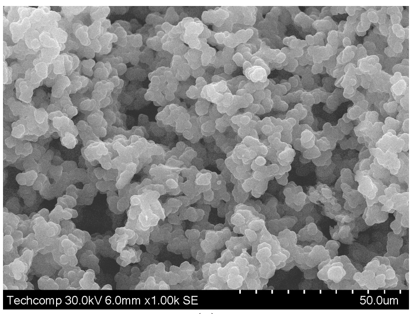
(b)



(c)



(d)



(e)

Fig. 1. SEM images of carbon aerogel

clear that the particle size of carbon aerogels increases with the increase of nR/nC at the same R_f mass concentration. This was because the active center quantity of the reaction systerm decreased with the catalytic reduced. As can be seen from Fig. 1, with the increase of nR/nC, namely catalyst dosage reduced, the partical size became larger. Though change the value of nR/nC, the aerogel structure can be controlled.

Bore diameter and surface aera: Fig. 2 shows the pore size distribution with different quality catalyst carbon aerogels. From 1# to 5#, the specific value of nR/nC were 500, 800, 100, 1200 and 1500. It can be seen that pore structure distribution range focused on 4 nm from 1# to 5#, but the pore volume were very different. When the specific value of nR/nC was 800, the pore volume reached maximum.

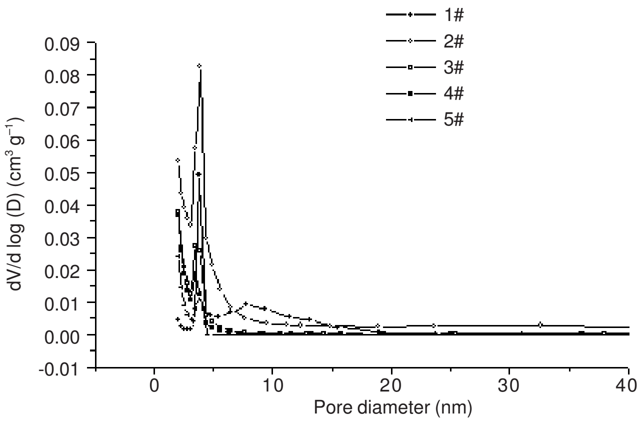


Fig. 2. Pore size distribution of carbon aerogels.

Table-1 listed the data from nitrogen adsorption measurements. It is very clear that the S_{BET} first increased and then decreased with the increasing of nR/nC. When nR/nC was 1000, the S_{BET} reached 1011.2 $m^2 g^{-1}$. Whe nR/nC was 800, the pore volume reached maximum value 0.665 $cm^3 g^{-1}$.

TABLE-1
SYNTHETIC CONDITIONS AND TEXTURE PARAMETERS OFCARBON AEROGELS

nR/nC	Catalyst	S_{BET} ($m^2 g^{-1}$)	V ($cm^3 g^{-1}$)
500		329.49	0.224
800		840.4	0.665
1000	Calvital	1011.2	0.428
1200		1000.2	0.415
1500		904.3	0.353

Electrochemical impedance spectroscopy measurements: As it can be seen in Fig. 3, using electrochemical AC impedance method, the Nyquist curves can be obtained made up of carbon electrolyte. Curves on the right are the low frequency aerea and on the left are high frequency aera. Linear part of the Nyquist reflected the linear relationship in the real part and imaginary part of the electrochemical systerm impedance. It also embodied the features of diffusion vacancy electrode process, the larger of the linear slope, the greater of the imaginary part impedance, which reflected the electrochemical systerm capacitive. From Fig. 3, the loop in the high frequency include ohm resistance and charge transfer resistance. In the right of the circular arc that the extendedsion cord intersected with the real part showed the electrochemical system impedance.

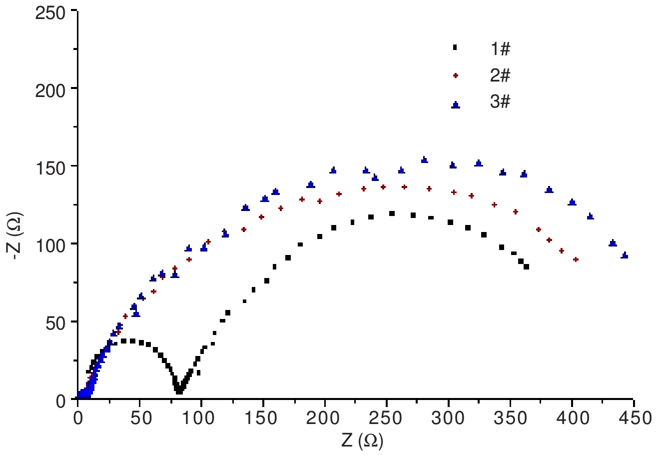


Fig. 3. AC impedance spectroscopies of carbon aerogel

Conclusion

A new type of carbon aerogel with high BET surface aera can be produced under ambient drying conditions instead of supercritical drying. The highest surface aera and pore volume of carbon aerogels produced are around 840 m^2/g and 0.665 cm^3/g, with 90 % of the pore volume as mesopores. The pore structure distribution and pore volume can be controlled by adjusting the quantity of catalyst.

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