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Facile Preparation of Carbon Aerogels with Different Drying Methods

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Abstract. Carbon aerogels were prepared through a polycondensation of phloroglucinol, resorcinol and formaldehyde with sodium carbonate as the catalyst under different drying methods. The drying methods were room temperature drying, vacuum drying and freeze drying. The effects of different drying methods on the porous structure of carbon aerogels were studied systematically. The texture and pore structure were investigated under scanning electron microscopy (SEM), transmission electron microscopy (TEM), X-ray diffraction (XRD) and a surface-area analyzer. The results show that the freeze-drying method was good for the specific surface area of carbon aerogels, up to 843 m² g⁻¹.

1. Introduction

Over the past several decades, carbon materials have gotten more and more attentions because of high-performance physical and chemical properties. Carbon aerogels exhibited high specific surface area and specific capacitance which are ideal materials for supercapacitors and batteries. A growing interest has been paid for carbon aerogels in recent years because of their excellent physical and chemical properties, such as porous properties, electric double-layer properties and low thermal conductivity.

Carbon aerogels were first synthesized through a sol-gel process under condensation-polymerization of resorcinol (R) and formaldehyde (F) using sodium carbonate (C) as catalyst with drying at room temperature. Currently, people often use freeze drying method and supercritical drying method to prepare dried aerogels. CAs have been investigated extensively and reported for use in next-generation energy storage applications in the use of their porous structure and excellent electric-double-layer capacitance behavior.

Organic solvent exchange and a lengthy of drying time are the drawbacks of the traditional drying methods. The dangers of high operational pressure exist in the process of supercritical drying. Freeze-drying method and vacuum drying method have some advantages: 1) length of the drying time; 2) improvement of the specific surface area; 3) increasing of the operational security.

Different drying methods have been applied for the synthesis of carbon aerogels in recent years. People use freeze drying method to prepare carbon aerogels, which can get short gel time and excellent porous structure. Meanwhile this method can well control the pore size distribution of carbon aerogels. In this paper, we prepared carbon aerogels through a sol-gel process under condensation-polymerization of phloroglucinol (P), R and F using C as catalyst with freeze-drying method, room temperature drying method and vacuum drying method, respectively. The effect of different drying methods on the physical structure is studied systematically, the specific surface area,



pore volume, pore size and structure properties of the carbon aerogels are investigated.

2. Experimental

2.1. Materials

The chemical reagents purchased are analytical grade: phloroglucinol, resorcinol, formaldehyde (30%), and sodium carbonate (C). Ultima IV X-ray diffraction instrument (XRD, Rigaku, Japan), Specific surface analyzer ASAP2420 (micromeritics, USA), scanning electron microscope (SEM), transmission electron microscopy (TEM, JEM-2100F, JEOL, Japan) and X-ray photoelectron spectroscopy (XPS, PHI5600 Physical Electronics).

2.2. Synthesis of the Carbon Aerogels

Using polycondensation of phloroglucinol (P), resorcinol (R) and formaldehyde (F) with sodium carbonate as catalysts (C) and deionized water as the solvent to prepare carbon aerogels. The molar ratio of P / R, (P + R) / F and (P + R) / C were 0.15, 0.5, and 800, respectively. The solution concentration was up to 45 wt%. P, R, F, and C were added into deionized water. We use ultrasonic oscillator to dissolve the raw materials to get a homogeneous solution in a tubes. Putting the tubes in a water bath and obtaining the hydrogels under 50°C for 8 h. The hydrogels were dried under freeze drying method, room temperature drying method and vacuum drying method, respectively. We carbonized the as – prepared aerogels at 900°C for 3 h and the heating rate was 2°C/min, which was processed under a flowing nitrogen atmosphere (100 mL/min).

3. Results and Discussion

We adopted the N₂ adsorption–desorption isotherms of different samples to analyze the porous structure and the results are presented in Fig. 1. The specific surface area (S_{BET}), micropore surface area (S_{micro}), BJH desorption average pore diameter (D_{average}) and total pore volume (V_{total}) of as-prepared CAs samples was shown in Table 1. As shown in figure 1, type-IV isotherms could be seen in all the N₂ adsorption–desorption isotherms of CAs, which indicates the as – prepared samples have an excellent porous structure. As seen in Table 1, CAs-FD exhibited the highest specific surface area, up to 843 m² g⁻¹, which is good for the transition of different ions in carbon aerogels. As seen in figure 2, a narrow pore size distribution could be clearly seen in all the samples and CAs-FD exhibited a narrower pore size distribution, 10nm - 20nm of the pore size range . This result indicated the freeze drying method was good for the prepared of pore size controlled carbon aerogels.

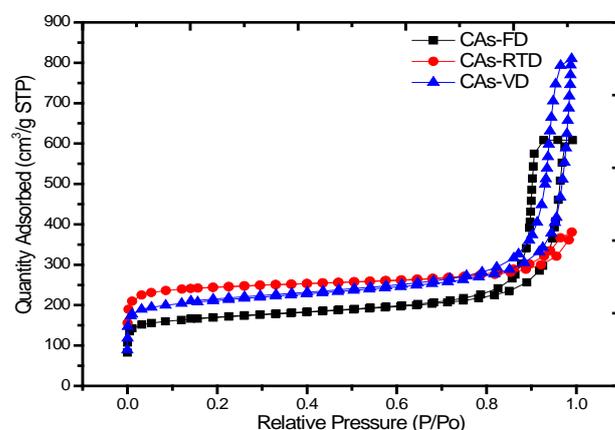
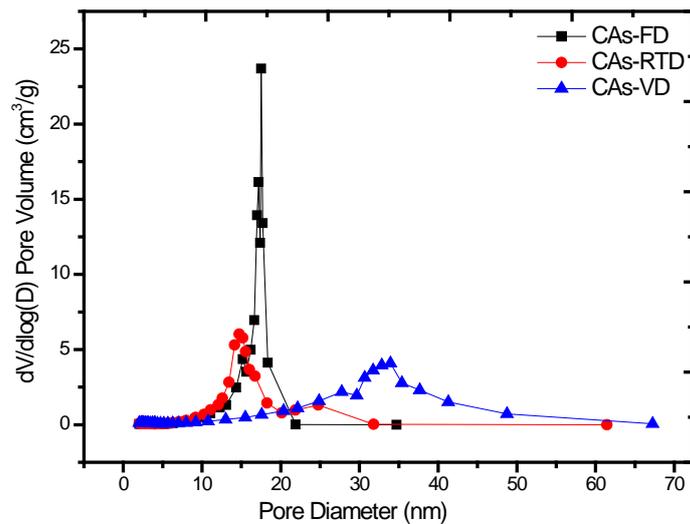


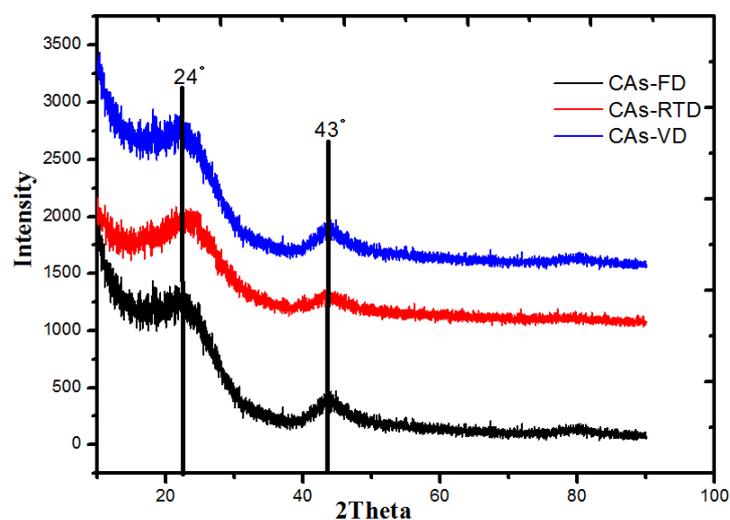
Figure 1. Nitrogen adsorption – desorption isotherms of different CAs samples (CAs-FD: under freeze drying method; CAs-RTD: under room temperature drying; CAs-VD: under vacuum drying)

Table 1. Textual properties of different CAs samples

Entry	S_{BET} (m^2/g)	S_{micro} (m^2/g)	D_{average} (nm)	V_{total} (cm^3/g)
CAs-FD	843	486	7.21	1.341
CAs-RTD	593	353	7.01	0.981
CAs-VD	765	426	8.53	1.607

**Figure 2.** Pore size distribution of different carbon aerogels (CAs-FD: under freeze drying method; CAs-RTD: under room temperature drying; CAs-VD: under vacuum drying).

We obtained the XRD patterns of different as – prepared samples samples, which are shown in Fig. 3. As presented in Fig. 3, a 2θ of 24° and 42° about two diffraction peaks were very clearly, which are attributed to the planes (002) and (101), respectively. This result shows that graphite carbon and amorphous composed carbon carbon aerogels.

**Figure 3.** XRD patterns of different carbon aerogels (CAs-FD: under freeze drying method; CAs-RTD: under room temperature drying; CAs-VD: under vacuum drying).

The SEM images of different samples were obtained, which are shown in Fig. 4. As shown in Fig. 4, an interconnected three-dimensional porous morphology could be seen in all the images. As seen in Fig. 4, the carbon aerogels have an obvious interconnecting network structure.

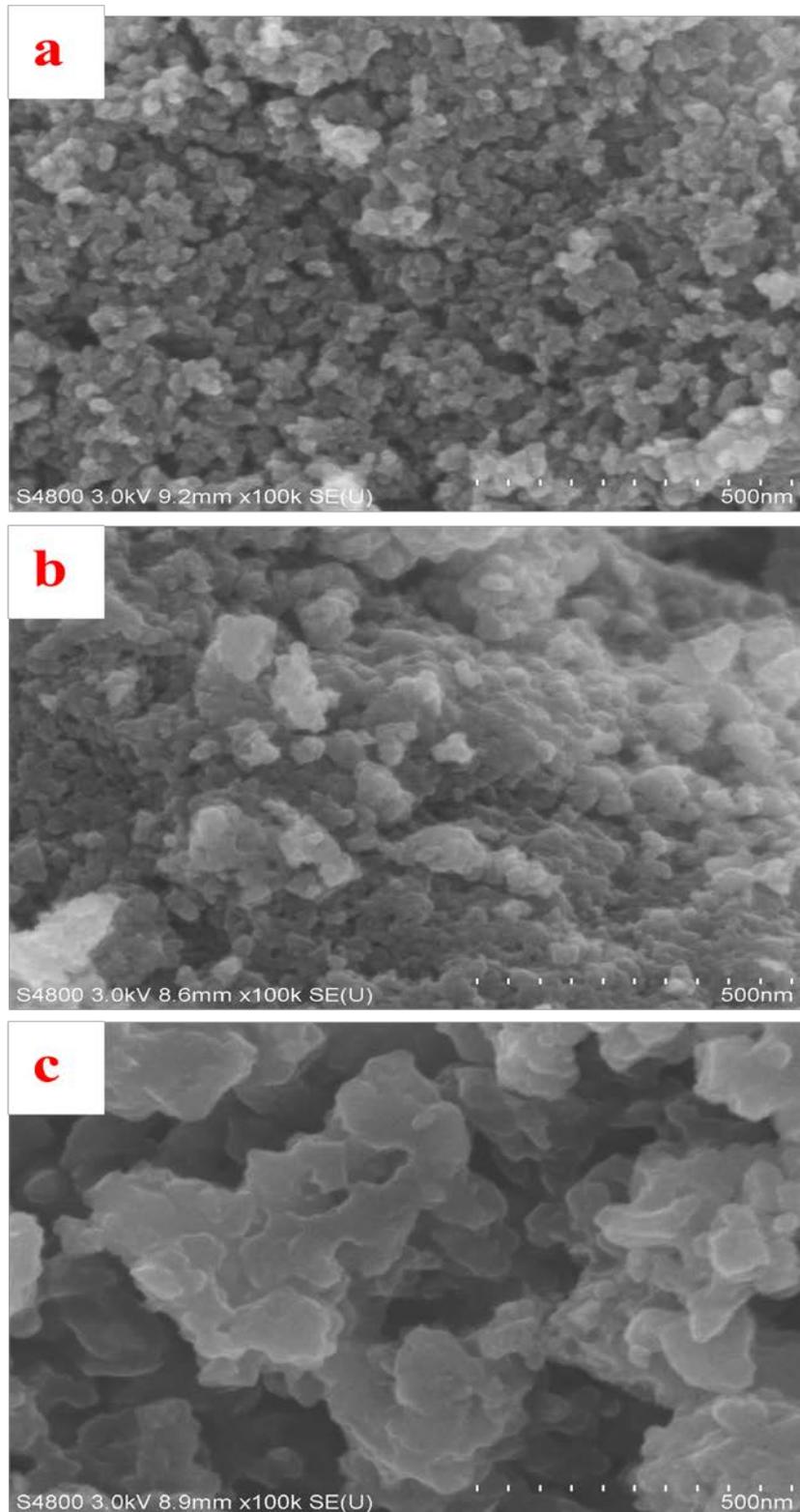


Figure 4. SEM images of different CAs (a: under freeze drying method; b: under room temperature drying; c: under vacuum drying).

4. Conclusion

Different carbon aerogels were prepared through polycondensation of P, R and F with using C as the catalyst under freeze drying method, room temperature drying method and vacuum drying method, respectively. The porous structure and electrochemical properties of as - prepared carbon aerogels were promoted through the freeze - drying method. A new avenue to obtain excellent carbon aerogels was found by different drying method and This materials show a widely applications of carbon aerogels.

5. Acknowledgments

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