Preparation of Porous Carbon Nanorods by Cellulose Template Method and Their Supercapacitor Properties

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Abstract. Aiming at the properties of carbon materials, this article synthesized porous carbon nanorod materials by cellulose template method and characterized them by various analysis methods. The results show that these porous carbon nanorod materials have unique morphology and excellent electrochemical performance. It can be seen that these porous carbon nanorod materials have potential application value in capacitors.

1. Introduction
Due to the difference between peak and trough periods of power consumption, a large amount of electrical energy is wasted, so people seek to use energy storage to solve this problem. However, current energy storage batteries still have some shortcomings in terms of energy density and cycle life. As a result, supercapacitors with more advantages have emerged. Current supercapacitors cannot fully meet actual needs in terms of electrode materials. It is a general trend to study new preparation methods to improve the performance of capacitors, and its theoretical and practical significance are extremely important.

2. Overview of the related theory
2.1. Super capacitor
Supercapacitors are composed of five parts, including positive electrode, negative electrode, electrolyte, current collector and diaphragm. It can be divided into electric double layer type and pseudocapacitive type. The basic principle of the former is to establish a solution/electrode system, so that the positive and negative ions on the electrode and in the solution attract each other to generate a relatively stable potential difference between the two stages, so that the electrode generates an equal amount of ions with opposite electrical properties within a certain distance. It can charge to keep it electrically neutral. The latter uses the principle of redox reaction or Faraday charge transfer reaction, and uses the chemical reaction process on and near the surface of the electrode material to store and release charge. At present, since electric double layer capacitors do not involve chemical reactions during the entire charging and discharging process and have a long cycle life, they are also more widely used[1]. The parameters that affect the performance of supercapacitors mainly include three aspects, including specific surface area, pore size distribution, and material conductivity.

2.2. Porous carbon material
Considering that carbon materials in the nanocrystalline form in the past are relatively expensive, in
today’s research, a biomass matrix is usually used to synthesize porous carbon materials. This material is porous with a highly developed pore structure. According to the size of the pore size, it can be divided into mesopores, micropores and ultramicropores. These materials have good electrical conductivity, hydrophobicity, and physical and chemical stability, and are suitable for the preparation of supercapacitors. If it needs to be widely used, this type of material still needs to be improved.

2.3. Template method
The template method has become one of the common preparation methods for porous carbon nanorod materials. It uses the template as the main structure to modify and control the shape, size and characteristics of the final material. Specifically, the template method can be divided into hard template method and soft template method according to the nature of the template used. The former is maintained by covalent bonds, while the latter is maintained by weak interactions between molecules. The advantage of the template method is that the structure of the material prepared by it has better controllability. The structural characteristics of the material are similar to that of the template. It has a larger specific surface area and total pore volume, and its performance and quality are more excellent[2].

3. Perorous carbon nanorodar materials are prepared using cellulose as template

3.1. Experimental reagents and experimental instruments
The main equipment used in this experiment includes constant temperature magnetic stirrer, tube furnace, electronic balance, electrochemical workstation, oil bath, field emission scanning electron microscope, etc. The drugs used include cellulose triacetate, potassium hydroxide, ethanol, isopropanol, dilute hydrochloric acid and dilute sulfuric acid.

3.2. Preparation of porous carbon nanorodar materials

3.2.1 Preparation of cellulose template.
It should weigh a certain amount of cellulose triacetate into a round-bottomed conical flask, heat it with a device with a circulating water cooling system, and then stir at low speed. After the stirring is completed, it can slowly inject an appropriate amount of isopropanol solution, and keep the temperature and stirring speed constant, and a more uniform solution can be formed after about 1 hour. Subsequently, it can use a pipette to transfer these homogeneous solutions to a polytetrafluoroethylene mold and cool to room temperature. During the cooling process, the solution will gradually turn into a gel state. After it turns into a gel state, it is placed in an acetone solution for multiple exchanges, and then dried to obtain the final cellulose template[3].

3.2.2 Preparation of porous carbon nanorod materials.
It should weigh a certain amount of cellulose template material, grind it with a masher, and then weigh a small amount of potassium hydroxide solid to make a solution. The crushed cellulose template material and the alcohol aqueous solution of potassium hydroxide are uniformly mixed in an ultrasonic environment. After the mixture is uniformly mixed, the mixture is heated in an oil bath for a period of time to remove the water and ethanol in the reaction system. The product obtained after drying is then placed in a tube furnace. High temperature calcination is carried out under the protection of nitrogen atmosphere. After the calcination is completed, the obtained product is etched with a 1mol/L dilute hydrochloric acid solution. After the etching is completed, centrifuge with water and ethanol for several times until the solution becomes neutral. Then the reaction system is placed in an oven and dried at a constant temperature of 60 ℃ to finally obtain a porous carbon nanorod material[4].

3.2.3 Main instruments are used for material characterization.
In this experiment, the main instruments used are Scanning Electron Microscope (SEM) and Transmission Electron Microscope (TEM) to observe the morphology and size of the prepared materials.
An X-ray diffractometer is used to observe the crystal structure of the prepared porous carbon nanorods. The specific gravity of each component in the composite material is quantitatively tested by using a thermogravimetric analyzer. High-power electron microscopy (HRTEM) and mapping tests are performed on materials by using field emission transmission electron microscopy. It can use infrared spectroscopy to study the molecular structure and chemical bonds of substances. The laser confocal Raman spectrometer can identify substances and analyze their properties. At the same time, in this experiment, Raman spectroscopy is used to analyze the degree of graphitization of carbon materials. It can also use X-ray electron spectrometer to analyze the surface of solid materials.

3.2.4 Assembly of electrode materials.
In this experiment, the electrochemical performance test for porous carbon nanorods is carried out at room temperature, and the test instrument uses CHI660C electrochemical workstation. The cyclic voltammetry (CV), constant current charge-discharge curve method, and AC impedance method commonly are used in this workstation for measurement. When preparing the working electrode, it should first weigh a small amount of sample and disperse it in a small amount of deionized water, and then ultrasonically treat the mixture to ensure that the mixture is highly dispersed. The dispersion liquid is then dropped on the working electrode. In this experiment, a glassy carbon electrode with a diameter of 3mm is used as the working electrode.

4. Supercapacitor properties of porous carbon nanorod materials

4.1. Appearance representation
The micromorphology of porous carbon nanorod materials prepared in this experiment is shown in figure 1.

![Figure 1. A SEM diagram of porous carbon nanorodar materials](image)

Figure 1. A SEM diagram of porous carbon nanorodar materials

It is not difficult to see from figure 1 that the porous carbon nanorod material prepared in this experiment has a better pore structure and the distribution of pores is relatively uniform. It can also be inferred that the elements such as O and S contained in it are also evenly distributed in the carbon nanorod material.

4.2. The porous nanorod materials
The XRD patterns and infrared characterization patterns of the porous nanorod materials prepared in this experiment are shown in figure 2 and figure 3.
According to the results in figure 2, it can be found that the structure of the material has changed significantly during the preparation process. After the reaction, the sample showed characteristic diffraction peaks of carbon at 13° and 30°, which proves that the carbon material formed after carbonization has an amorphous structure\(^5\).

At the same time, according to the results in figure 3, it is not difficult to see that the infrared absorption peak of the carbon-carbon double bond is at 1620 cm\(^{-1}\), while the absorption peak of the alkyne bond is not observed at 2100 cm\(^{-1}\). At the same time, the water peak at 3300 cm\(^{-1}\) disappears, and the peak greater than 3500 cm\(^{-1}\) is the hydroxyl absorption peak of the carboxylic acid.

4.3. The XPS test results of the sample
The XPS spectroscopy of porous carbon nanorods prepared in this experiment is shown in figure 4.
Figure 4. The XPS spectrum diagram of the sample

It can be seen from the XPS spectrum of the sample that the porous carbon nanorod material synthesized in this experiment only contains C, O and S. The peaks of these three elements correspond to 284.9, 532.0, and 168.3 eV, respectively. During the reaction process, other metal ions and other impurity elements have been basically removed, which indicates that the carbonization effect of the material is more obvious. The content of carbon-carbon double bonds is also high, which indicates that the XPS analysis results of the samples are consistent with the infrared characterization analysis results in figure 4.

4.4. Nitrogen desorption adsorption test of sample

In the electrochemical field, the specific surface area is the most important factor affecting the performance of supercapacitors. In order to determine the specific surface area and pore size distribution curve of the prepared material, the sample is tested for nitrogen adsorption and desorption. The nitrogen adsorption and desorption isotherm of the sample and the DFT pore size distribution curve are shown in Table 5 and Table 6.

Figure 5. Nitrogen adsorption and isothermal of the sample

Figure 6. The DFT aperture distribution curve of the sample
As can be seen from figures 5 and 6, the samples prepared in this experiment showed H4 hysteresis loops during the increase in relative pressure, indicating the existence of a cracked mesoporous structure and a relatively large amount of adsorption. This shows that there are a large number of microporous structures in the sample, and the potassium hydroxide used in this experiment also plays a role in activating the material, increasing the specific surface area of the material, and the larger specific surface area of the material can provide more activity site, increase the energy storage of the material.

4.5. Electrochemical performance test of samples
In the electrochemical performance test, the electrolyte is still a 1mol/L dilute sulfuric acid solution, and the scanning rate is 0.06V/s. After the scanning process is over, the obtained curve is shown in figure 7.

![Figure 7. Specific capacitance curve of the sample](image)

Figure 7. Specific capacitance curve of the sample

It can be seen from Figure 7 that the porous carbon nanorod material prepared in this experiment has better performance. Its specific capacitance is relatively high, even at a higher current density, its specific capacitance value is still high, and it is expected to have a high practical application value. At the same time, the electrochemical cycle stability of the material is relatively good.

5. Conclusion
In the experiments in this article, porous carbon nanorod materials are prepared by using cellulose as raw material, calcination at high temperature and activation with potassium hydroxide. Through the analysis of the materials, it can be known that the prepared carbon material has a uniform porous structure, and the prepared carbon material has a larger specific surface area and excellent electrochemical performance, which is of great significance to the environment and life. Obviously, carbon materials prepared by the cellulose template method have broad application prospects in supercapacitor energy storage devices.

References
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