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Synthesis and electrochemical capacitance of mesoporous $Co(OH)_2$

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Abstract

Mesoporous $Co(OH)_2$ was synthesized by using $CH_3(CH_2)_{10}CH_2OSO_3Na$ as soft template and urea as hydrolysis-controlling agent. The composition and microstructure of $Co(OH)_2$ was investigated by X-ray diffraction spectroscopy (XRD), scanning electron microscopy (SEM) and nitrogen adsorption and desorption isotherms. Structure characterizations indicated a good mesoporous structure for the prepared $Co(OH)_2$ with adsorption average pore diameter of 11 nm and Brunauer–Emmett–Teller (BET) specific surface area of $283\,m^2\,g^{-1}$. Cyclic voltammogram, galvanostatic charge–discharge test, and electrochemical impedance spectroscopy (EIS) analysis showed that the $Co(OH)_2$ possessed good capacitive behavior. The maximum specific capacitance of $341\,F\,g^{-1}$ was obtained for the mesoporous $Co(OH)_2$ at a charge/discharge current density of $5\,mA\,cm^{-2}$.

Keywords: $Co(OH)_2$; Mesoporous; Electrochemical capacitance

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1. Introduction

Electrochemical capacitors have recently attracted great attention because of their high capacitance, potential applications in electronic devices, and their possibility of coupling with batteries to provide pulses of peak power during acceleration and on uphill gradients [1,2]. More interest has been aroused in two types of supercapacitors, the double-layer supercapacitors and redox supercapacitors with different charge-store modes. In the former case, the capacitance is derived from charge separation in the carbon-based materials. In the latter, a Faradaic process takes place due to redox reactions based on metal oxides and electronically conducting polymers [3]. On account of the low utilization of the carbon-based material [4] and degradation of the conducting polymer material as a result of swelling and shrinkage of electroactive polymers during cycling [5,6], substantial research efforts have been focused on identifying more suitable electrode materials for supercapacitors. One promising approach is the use of transitional metal oxides for supercapacitors. The ability to achieve high specific capacitance of materials like RuO_2 and IrO_2 [7] has spurred research in this field. However, the high cost and toxic nature of these materials has necessitated the need to develop alternative electrode materials with improved characteristics and performance. Electroactive material $Co(OH)_2$ as a candidate has been demonstrated to achieve good electrochemical capacitance performance [8].

Since electric double-layer capacitance and pseudocapacitance due to the redox reaction are both interfacial phenomena, the materials for electrochemical capacitors should possess a high specific surface area with a suitable pore-size distribution to enhance the charge-storage capacibility [9]. Thus, the control of the surface morphology of the electrode material as the most important design parameter is the next logical strategy. Recently, the extension of the surfactant templating procedure to the formation of non-silica mesoporous oxide with higher specific surface area and proper pore-size distribution has been great widespread and attracted great interest [9–14]. Such unique mesoporous system and large specific surface area of the electroactive materials are expected to favour the ion transfer in the pore system and increase the electroactive material–electrolyte interface area, which is beneficial to the improvement on the electrochemical performance.

A rational idea inspired by the above-mentioned aspects is to create of porous structure and channels as a natural way to improve the electrochemical performance of the electrode materials. Herein, our strategy is to use anionic surfactant $CH_3(CH_2)_{10}CH_2OSO_3Na$ as a soft template, urea as a hydrolysis agent to synthesize mesoporous $Co(OH)_2$, all the electrochemical tests demonstrate that the as-prepared mesoporous

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$Co(OH)_2$, with a high specific area of 283 $m^2\,g^{-1}$, possesses good electrochemical capacitance performance.

2. Experimental

2.1. Synthesis and characterization of the mesoporous $Co(OH)_2$

All chemicals used in this work were of analytical grade reagents used without further purification. The synthesis protocol adopted in the paper was described as follows: $CoCl_2{\cdot}6H_2O$, $CH_3(CH_2)_{10}CH_2OSO_3Na$, urea and double-distilled water were taken in the mole ratio of 1:2:30:80. The mixture was stirred at 40 °C for 2 h to yield a transparent solution, then heated to 80 °C and maintained at such temperature for 2.5 h. The resulted mixture was cooled to the room temperature to restrain the further hydrolysis of urea. The product of the reaction was filtered, washed repeatedly with distilled water, and then dried in the air. To remove the excess surfactant, the solidified product was abraded, mixed with a 0.05 mol ethanol solution of sodium acetate and stirred at 40 °C for 1.5 h. Finally, the product was washed with copious ethanol and then dried at 50 °C. The morphology of the sample was examined by SEM (LEO 1430VP, Germany). The X-ray diffraction patterns of the samples were observed by Mac 18 XCE, using a Cu Kα source. The Brunauer–Emmett–Teller (BET) surface area and pore volume of the prepared $Co(OH)_2$ were obtained with a Micromeritics ASAP2010 system. Infrared spectra were recorded with a model 360 Nicolet AVATAR FTIR spectrophotometer.

2.2. Electrochemical tests

Electrodes for electrochemical capacitors were prepared by mixing the active materials with acetylene black (AB) and PTFE (polytetrafluoroethylene) (the weight ratio of $Co(OH)_2$:AB:PTFE = 7.5:2:0.5). A small amount of 1 M KOH solution was then added to form a more homogeneous mixture, which was pressed at 1.2×10^7 Pa on nickel grid treated with HCl solution. Each electrode contained 7.5 mg of electroactive material $Co(OH)_2$. All electrochemical measurements were carried out in 1 M KOH in a half-cell setup configuration at room temperature. A platinum gauze electrode and a standard calomel electrode (SCE) served as a counter electrode and reference electrode, respectively. Electrochemical performance was evaluated by cyclic voltammetry (CV) and electrochemical impedance analysis on CHI660 electrochemical workstation. The galvanostatic charge/discharge of the electrodes was evaluated with Arbin BT2042 battery workstation system in a certain range of potentials.

3. Results and discussion

3.1. The XRD pattern and FTIR spectroscopy for the prepared $Co(OH)_2$

The XRD pattern of the mesoporous $Co(OH)_2$ is shown in Fig. 1, and all the peaks can be indexed to the diffraction data of the spinel structure $Co(OH)_2$ (JCPDS, No. 74-1057). To further demonstrate the complete removal of surfactant $CH_3(CH_2)_{10}CH_2OSO_3Na$ and the existence of $Co(OH)_2$, FTIR spectroscopy was applied to provide additional evidence. The FTIR spectra of the prepared $Co(OH)_2$ and $CH_3(CH_2)_{10}CH_2OSO_3Na$ are shown in Fig. 2. From the comparison between the spectrum (a) and (c), it is clear that, in the spectrum of $Co(OH)_2$, the prominent bands at 2850–3000 and 1370–1380 cm^{-1} for typical C–H symmetric stretching and bending vibrations corresponding to the alkyl tails of the surfactant $CH_3(CH_2)_{10}CH_2OSO_3Na$, respectively, disappears after extensive leaching with copious ethanol, confirming the complete removal of surfactant $CH_3(CH_2)_{10}CH_2OSO_3Na$. From the spectrum (b), it is evident that the existence of corresponding Co–O stretching mode at 440 and 490 cm^{-1} [15], respectively, and a strong stretching vibration of O–H at 3670 cm^{-1}.

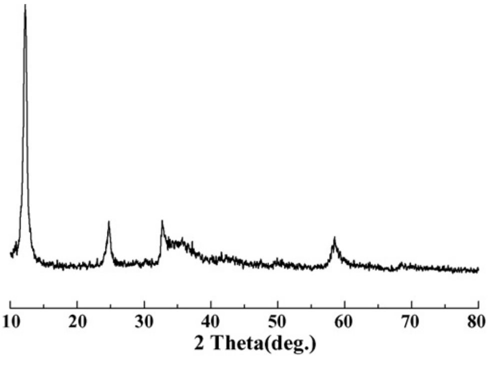

Fig. 1. XRD pattern of mesoporous $Co(OH)_2$.

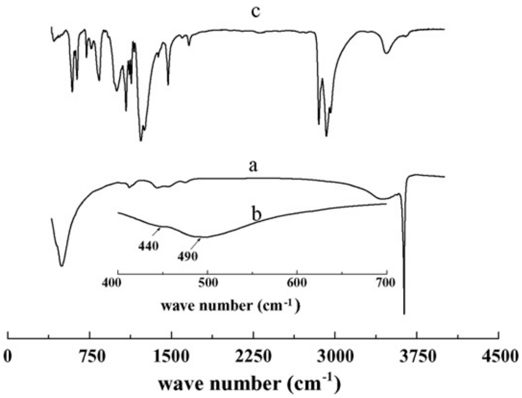

Fig. 2. FTIR spectra of the prepared $Co(OH)_2$ (a, b) and $CH_3(CH_2)_{10}CH_2OSO_3Na$ (c).

3.2. The morphology character of the mesoporous $Co(OH)_2$

Fig. 3 shows the SEM image for the prepared mesoporous $Co(OH)_2$. It is obvious that the 200–300 nm $Co(OH)_2$ particles appear to possess a most loosely packed structure and many pores with ca. 10 nm pore size. To obtain detailed information about pore size and specific surface area, static

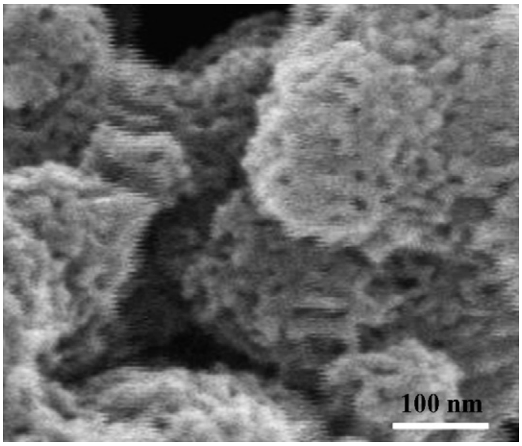

Fig. 3. SEM of mesoporous $Co(OH)_2$.