

Short Communication

Synthesis of Nickel Oxide/Active Carbon and Electrochemical Performance

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NiO/AC particles were synthesized by infinite dilute method. The synthesized NiO/AC particles with a structure of cubic crystalline phase are well dispersed and bulk like. The electrochemical tests show that the NiO/AC particles have relatively high capacitance and excellent capacitive retention. The good structure and excellent performance suggest its promising application in supercapacitors.

Keywords: infinite; bulk; crystalline

1. INTRODUCTION

In recent years, electrochemical supercapacitors (ES) or ultracapacitors have attracted significant attention, mainly due to their high power density, long cycle life, and bridging function for the power/energy gap between traditional dielectric capacitors (which have high power output) and batteries/fuel cells (which have high energy storage) [1-4]. Transition metal oxides are a class of important mineral materials that have drawn extensive research attention as electrode materials. Transition metal oxides such as ruthenium oxide [5], manganese oxide [6-8], cobalt oxide [9], and nickel oxide [10-12] are qualified to be electrode materials for electrochemical capacitor [13]. Some of them, such as ruthenium oxide and iridium oxide, exhibit excellent properties for use as pseudo-capacitive electrode materials [14]. Nevertheless, it is also noted that the materials of this class are rare and expensive. Among these materials, nickel oxide (NiO) attracts particular interest due to its high specific capacitance, high chemical/thermal stability, practical availability, environmentally benign nature and lower cost than others [15-18].

In this work, we report a facile approach to synthesize NiO/AC by infinite dilute method. The NiO/AC particles have a bulk structure which is believed to be very suitable for the electrode materials of supercapacitors.

2. EXPERIMENTAL

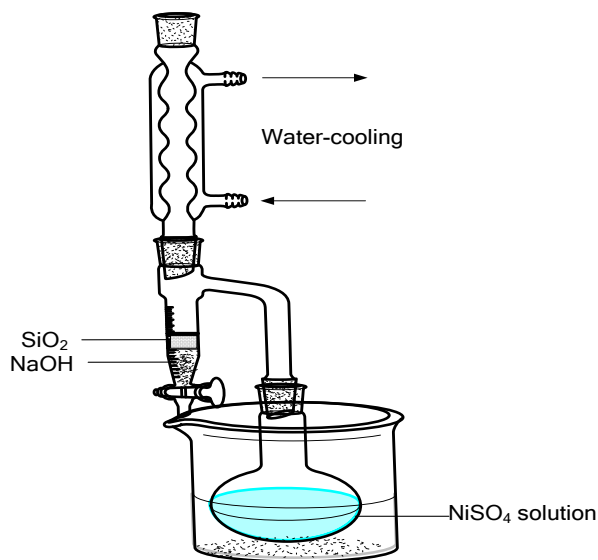


Figure 1. Schematic of apparatus for synthesis

2.1 Materials preparation

All the chemicals were of analytical reagent and used as received without further purification. In a typical preparation progress, 3.9 g of $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$ was dispersed into a solution containing 30 mL of de-ionized water (5 mL of Polyethylene glycol as dispersing agent), and then joined it and the active carbon (AC) in the flask. 1.2 g NaOH (AR) was added in the place as the Fig. 1, and above it was 1.2 cm height of quartz sand. Then, put the flask in the beaker filled with water. Heat the beaker at 100 °C for 48 h. The mixture was aged for an additional 3 h and then filtered, washed with distilled water. The product was obtained by calcination at 300 °C for 2 h under the protection of nitrogen at ramping rate of 2 °C min^{-1} .

2.2 Materials characterization

Powder x-ray diffraction (XRD) patterns of the sample was recorded on a Philips X'pert diffractometer with Cu $K\alpha$ radiation ($\lambda=1.5418 \text{ \AA}$). The morphology and the structure of the sample were examined with a field-emission scanning electron microscopy (FESEM, JEOL JSM-6300F).

2.3 Electrochemical measurements

The working electrode was prepared by 85 wt% of the active material (the proportion of NiO/AC is 1:1 and mixing uniformity), 10 wt% of conducting agent (carbon black), and 5 wt% of binder (polyvinylidene difluoride, PVDF). This mixture was pressed onto the glassy carbon electrode (Aida Hengsheng Technology co. td, Tianjin, China) and then dried at 60 °C. The electrolyte used was 3 M KOH aqueous solution. The capacitive performance of the sample was tested on the CHI608A electrochemical workstation with cyclic voltammetry and chronopotentiometry functions using three-electrode system (a saturated calomel electrode (SCE) as the reference electrode, a Pt electrode as the counter electrode).

3. RESULTS AND DISCUSSION

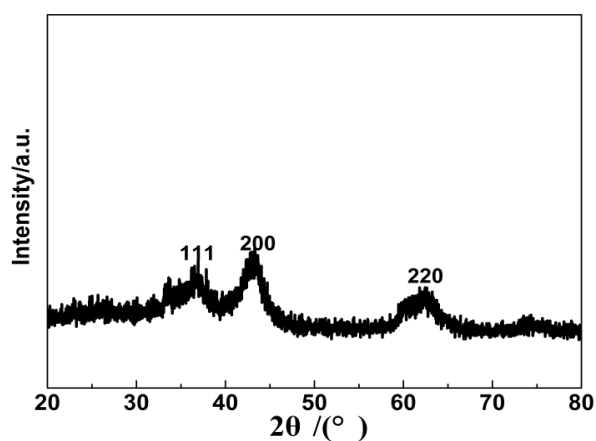


Figure 2. The XRD pattern of the sample NiO

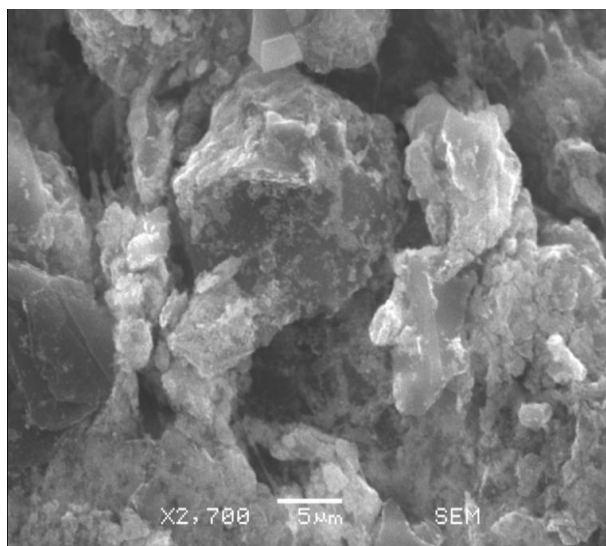


Figure 3. The SEM image of NiO particles

The crystal phase of the sample was analyzed by X-ray diffraction (XRD). The powder XRD pattern of the sample in our experiment is shown in Fig. 2. The characteristic peaks are at $2\theta = 37.3^\circ$, 43.3° and 62.9° [corresponding to (111), (200) and (220) reflection, respectively]. The sample is phase-pure NiO, all the identified peaks of which can be assigned to the cubic phase of NiO (JCPDS file No.4-835, Space Group: Fm $\bar{3}$ m, $a_0=4.1769 \text{ \AA}$). It means that we obtain the finer size and amorphous state of electrode materials to provide a large amount of superficial electroactive species to participate in faradic redox reaction. To further investigate the NiO/AC particles, SEM was adopted. Fig. 3 shows the SEM image of NiO/AC particles. It reveals that the synthesized NiO/AC particles which consist of the aggregated flakes are dispersed and bulk like.

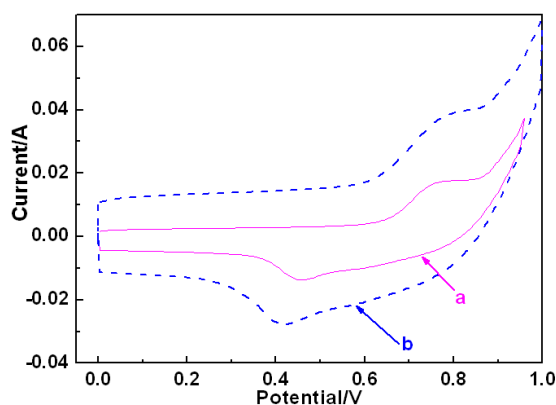


Figure 4. CV curves of the nickel oxide electrode at different scan rates (a=1 mV/s; b= 2mV/s)

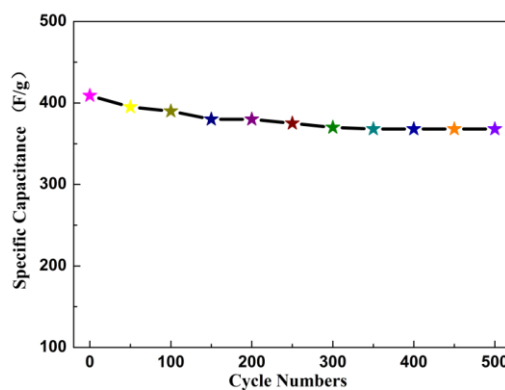


Figure 5. Cycle-life curves of NiO/AC electrode samples at 10 mA

Fig. 4 shows the CV curves of the NiO/AC electrode at different scan rates (a=1 mV/s; b=2 mV/s). The potential span is from 0 to 1.0 V (vs.SCE) in 3 M KOH aqueous solution. We can see that the CV curves have two intense peaks. One peak is anodic during the oxidizing reaction of Ni²⁺ to Ni³⁺, and the other is cathodic during the reverse process. These peaks come from fast and reversible redox processes that occur at the surface of the electrode, according to the following equation: $\text{NiO} + \text{OH}^- \rightleftharpoons \text{NiOOH} + e^-$. From the CV curves at scan rates of 1 and 2 mV/s, the corresponding specific

capacitances are 808 and 780 F/g respectively. Therefore, at a slow scan rate, full utilization of the electroactive surface of NiO particles enhances the specific capacitance. Fig. 5 shows the profile of the specific capacitance versus charge/discharge cycle numbers at 10 mA. At the beginning, the specific capacitance of NiO/AC electrode is 408 F/g. At the end of 500 cycles, the specific capacitance of NiO/AC electrode is 368 F/g which indicates that 90 % of their initial capacitance can be retained. The results suggest that these particles have relatively higher capacitance and more excellent capacitive retention than other articles [19].

4. CONCLUSIONS

In summary, NiO/AC particles have been prepared by a simple and cost-effective approach. With NaOH as precipitation agent and NiSO₄·6H₂O as raw material, NiO/AC particles were synthesized by infinite dilute method. X-ray diffraction (XRD) shows that the material is the NiO with a structure of cubic crystalline phase. Scanning electron microscope (SEM) shows that the synthesized NiO/AC particles are well dispersed and bulk like. When evaluated for supercapacitive performance, the specific capacitance is 408 F/g at 10 mA. With the increase of cycling times, the specific capacitance decreases slightly. 200 cycles later it can be retained at 368 F/g (90% of the initial capacitance). The electrochemical measurements reveal that this kind of unique NiO/AC material manifests pseudo-capacitive properties with high capacitance and good capacitance retention, which probably benefits from the bulk like structural feature. The good structure and excellent performance suggest its promising application in supercapacitors.

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