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Short Communication

A Novel Synthesis of Nickel Oxide and Its Electrochemical Performances

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The NiO particles were synthesized by repeated immersion method. The as-prepared particles are NiO, which are well dispersed and have a wave-like shape. The electrochemical tests show that the particles have relatively high capacitance and excellent capacitive retention. The good structure and excellent performance suggest its promising application in supercapacitor.

Keywords: novel, nickel oxide, electrochemical

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) [1-3]. 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, manganese oxide, cobalt oxide, and nickel oxide are qualified to be electrode materials for electrochemical capacitor [4-6]. Some of them, such as ruthenium oxide and iridium oxide, exhibit excellent properties for use as pseudo-capacitive electrode materials [7-9]. 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 [10-15].

In this work, we report a facile approach to synthesize NiO by repeated immersion method. The NiO particles have a wave-like shape which is believed to be very suitable for the electrode materials of supercapacitors. These particles were characterized by XRD, SEM. The supercapacitive behavior of NiO was investigated by cyclic voltammetry in 3M KOH electrolyte. The effect of scan rate on the supercapacitance of nickel oxide thin film has been investigated.

2. EXPERIMENTAL

2.1 Materials preparation

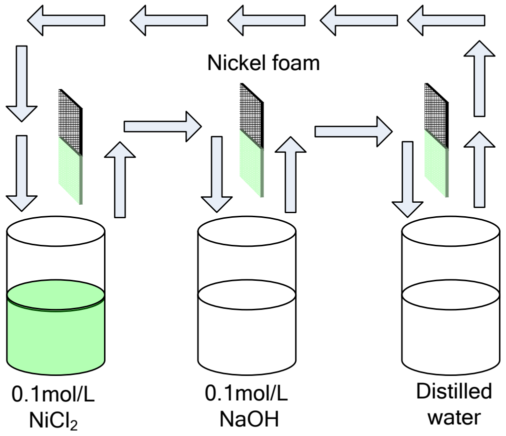

Figure 1. Schematic of apparatus for synthesis

The schematic representation of repeated immersion method chemical method is shown in Fig. 1. All the chemicals were of analytical reagent and used as received without further purification. Three breakers system is used for the deposition onto the nickel foam. In a typical preparation progress, the well cleaned nickel foam was dipped in 0.1 M NiCl$_2$ solution for 5 s. After that, we dipped the nickel foam in the 0.1 M NaOH solution for 5 s. Then, we dipped the nickel foam in the distill water for 5 s. Thus one cycle of immersion is completed. This cycle was repeated 50 times to increase the thickness of nickel oxide particles.

2.2 Materials characterization and measurement

Nickel oxide was characterized by X-ray diffraction (XRD), scanning electron microscopy and transmission electron microscopy. Powder x-ray diffraction (XRD) patterns of the sample was recorded on a Philips X'pert diffractometer with Cu Kα radiation (λ=1.5418 Å). 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 (Nickel oxide), 10 wt% of conducting agent (carbon black), and 5 wt% of binder (polyvinylidene difluride, 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 CHI660 electrochemical workstation (CHI, USA) 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). Experiments were carried out at room temperature. Powder x-ray diffraction (XRD) patterns of the sample was recorded on a Philips X'pert diffractometer with Cu Kα radiation (λ=1.5418 Å). The morphology and the structure of the sample were examined with a field-emission scanning electron microscopy (FESEM, JEOL JSM-6300F).

3. RESULTS AND DISCUSSION

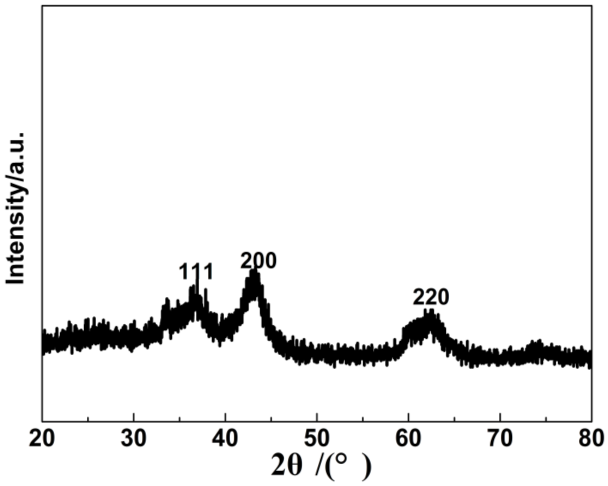

Figure 2. The XRD pattern of the sample NiO

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 2θ = 37.3°, 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: Fm3m, ao=4.1769 Å). 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.

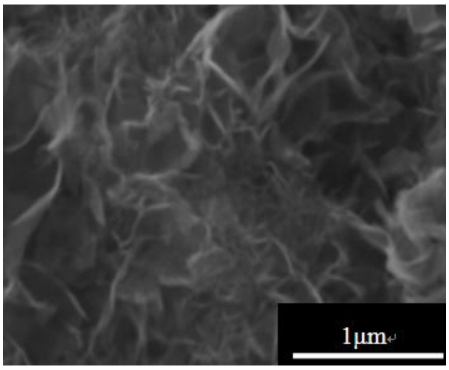

Figure 3. The SEM photograph of the sample NiO

Fig 3 shows the SEM image of the NiO. It reveals that the synthesized NiO particles are well dispersed. The structure has a wave-like shape which consists of the aggregated flakes.

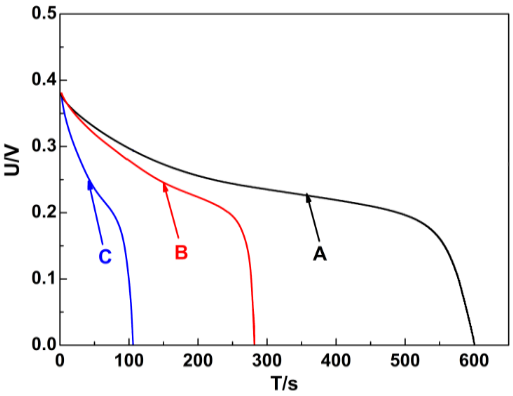

Figure 4. Discharge curves of NiO at different discharge current (A=5mA;B=10mA;C=20mA)

Fig.4 shows the discharge curves of NiO electrode at various current rates. It is very clear that there is a distinct plateau in every curve, corresponding to the Faradaic discharging process. The specific capacitance can be calculated according to the following equation:

$$C_m = I * \Delta t / (m * \Delta U) \quad (1)$$

where C_m (F/g) is the specific capacitance, I (A) is the discharge current, Δ t (s) is the discharge time, ΔU (V) is the potential range during discharge, and m (g) is the mass of the active material. Thus, the specific capacitance is calculated to be 525, 455 and 380 F/g at discharge current of 5, 10 and 20 mA, respectively. The decrease in specific capacitance is attributed to the NiO nanostructure that cannot sustain the redox reactions completely at a higher current. Therefore, at a lower current, full utilization of the electroactive surface of NiO enhances the capacitance.

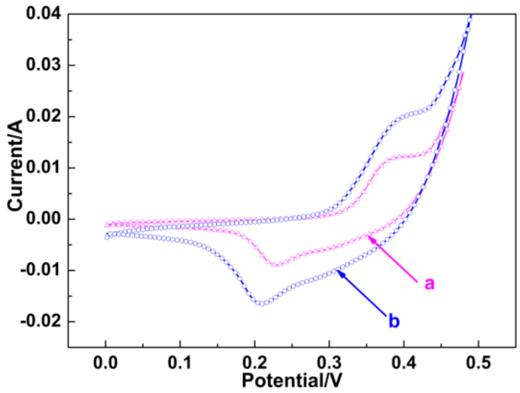

Figure 5. CV curves of the NiO at different scan rates (a=1mV/s; b=2mV/s)

Fig. 5 shows the CV curves of the NiO electrode at different scan rates (a=1 mV/s; b=2 mV/s). The potential span is from 0 to 0.5 V (vs.SCE) in 3 M KOH aqueous solution. We can see that the CV curves have two intense peaks. One peak is anodic (positive current density) during the oxidizing reaction of Ni^{2+} to Ni^{3+}, and the other is cathodic (negative current density) 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: $NiO + OH^{-} \leftrightarrow NiOOH + e^{-}$ [16-19]. From the CV curves at scan rates of 1 and 2 mV/s, the corresponding specific capacitances are 628 and 586 F/g respectively. Therefore, at a slow scan rate, full utilization of the electroactive surface of NiO particles enhances the specific capacitance. The decrease in specific capacitance is attributed to the NiO nanostructure which cannot sustain the redox reactions at a higher current.

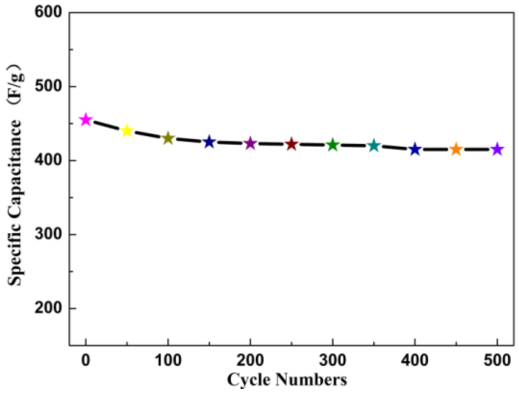

Figure 6. Cycle-life curves of NiO samples electrode

Fig. 6 shows the profile of the specific capacitance versus charge/discharge cycle numbers at 10 mA. At the beginning, the specific capacitance of NiO electrode is 455 F/g. At the end of 200 cycles, the specific capacitance of NiO electrode is 415 F/g which indicates that 91.2 % of their initial capacitance can be retained. The results suggest that these NiO particles have relatively high capacitance and excellent capacitive retention. The results suggest that these NiO particles have relatively higher capacitance and more excellent capacitive retention than other articles [21-23].

4. CONCLUSIONS

In summary, With NaOH as precipitation agent and $NiCl_2$ as raw material, $Ni(OH)_2$ particles were synthetized by repeated immersion method and sintered at 300℃ for NiO. X-ray diffraction (XRD) shows that the material is cubic crystalling phase of NiO; scanning electron microscope (SEM) shows the NiO particles present a wave-like shape. When evaluated for electrochemical performance, the NiO particles demonstrate improved electrochemical properties with a high capacitance of 455F/g at the current of 10mA. With the increase of cycling times, the specific capacitance decreased; after 200 times it can be retained at 415F/g (91.2% of the initial capacitance).

ACKNOWLEDGEMENTS
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