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Bhuvaneshwari R.S

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Research Article

EFFICIENT SYNTHESIS OF BINARY SPINEL $NICO_2O_4$ NANOPARTICLES FOR SUPER CAPACITOR APPLICATION

Bhuvaneshwari R.S*

Mahendra Arts and Science College, Kalippatti, Namakkal District 637501, Tamil Nadu, India

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ABSTRACT

The binary spinel nickel cobaltite ($NiCo_2O_4$) nanoparticles were successfully synthesized using Ni $(No_3)_2.6H_2O$, Co $(No_3)_2.6H_2O$ and NaOH by co-precipitation method. The synthesized samples have been systematically characterized by X-ray diffraction spectroscopy (XRD), Fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM) respectively. The spinel nickel cobaltite confirmed by XRD and FTIR analysis. Morphology have been in depth characterized by SEM. Electrochemical properties of Nickel Cobaltite are investigated using cyclic voltammetry (CV), which exhibit 225 Fg^{-1} at a scan rate 5 mV s^{-1}. The cost effective $NiCo_2O_4$ will be potential candidate for next generation of energy storage devices.

INTRODUCTION

The key technology challenges for 21st century scientists are global warming, climate change, and depleting of fossil fuels for the recent few decades. To solve these problems, there is an urgent need to development alternative energy storage devices with advanced energy storage and power density, like batteries and supercapacitors [1]. Generally, electrochemical capacitors (EC) or supercapacitors are considered as one of the potential candidates for high power energy storage devices and have attracted significant attention due to their high power density, long cycle life, and low maintenance cost [2, 3] As such, supercapacitors can replace or complement batteries in many applications, ranging from hybrid electric vehicles to pulse-power applications in the aerospace and defense industries. Several kinds of materials have been successfully used for electrochemical supercapacitor electrodes, such as carbon-based materials, metal oxides, and conducting polymers [4, 5]. RuO_2 is one of the best electrode materials for supercapacitor devices due to high specific capacitance, reversible charge-discharge features, and good electrical conductivity, all of which make it the focus of research and enhancement of supercapacitors having the potential to achieve higher energy and power densities [6-8]. However, the high cost and toxic nature of RuO_2 have impeded its commercial applications. Therefore to replace this ruthenium oxide, spinel $NiCo_2O_4$ has been considered to be one of the most attractive materials in terms of its high specific capacitance, low cost, natural abundance and environmental benignity [9]. The binary spinel nickel cobaltite ($NiCo_2O_4$) is one of the most promising materials with higher electronic conductivity by two orders greater than that of monometallic nickel oxides or cobalt oxides [10, 11]. These attractive features are more useful to develop high performance electrochemical supercapacitor applications. various strategies has been prepared to nickel cobaltite nanoparticles, such as spray pyrolysis, hydroxide decomposition route, combustion methods, pulsed laser depositions, and reverse micellar route etc., [12] among them co-precipitation method is one of the simple, cost effective, low temperature process and environmental benignity.

In this work, the nickel cobaltite nanoparticles were successfully prepared by co-precipitation method and then calcined at a moderate temperature of 400°C. The sodium hydroxide is crucial role to the formation of nickel cobaltite. The phase purity and crystallinity of obtained nanoparticles were characterized by XRD. The function groups and morphological studies of $NiCo_2O_4$ were successfully characterized by FTIR and SEM. Finally the electrochemical studies exhibited high specific capacitances.

Corresponding author: **Bhuvaneshwari R.S*
Mahendra arts And Science College, Kalippatti, Namakkal District 637501 Tamil Nadu, India

Experimental procedure

Ni $(No_3)_2.6H_2O$, Co $(No_3)_2.6H_2O$ and NaOH were used for this study. All chemicals were analytical grade and used without further purification. An appropriate quantity of Ni $(No_3)_2.6H_2O$ and Co $(No_3)_2.6H_2O$ were dissolved separately in 50ml distilled water to form homogeneous solution. Appropriate quantity of sodium hydroxide was added drop wise under constant magnetic stirring for four hour at 90 °C. The obtained precipitate was filtrated, washed with ethanol and distilled water to remove unexpected ions. Then dried at 100°C for 3 h at atmospheric pressure. Finally the dried sample was calcined at 400 °C for 4 h to obtain $NiCo_2O_4$ nanoparticles.

Material characterization

The crystalline phase of nickel cobaltite nanoparticles were analyzed by X-ray diffraction (XRD) measurement which was carried out at ambient temperature by using X'PERT-PRO diffractometer system (scan step of 0.05° (2θ), counting time of 10.16 s per data point) equipped with a Cu tube for generating Cu Kα radiation (kα = 1.5406 Å); as an incident beam in the 2-theta mode over the range of 10°–80°, operated at 40 kV and 30 mA. The Fourier transform infrared (FTIR) spectra were carried out using SHIMADZU-8400 FTIR spectrometer in the range of 4000-400 cm^{-1}. The morphology of the product was observed by scanning electron microscopy (SEM; JEOL-JSM-56100) operating under 20 kV accelerating potential.

Electrochemical characterization

The electrochemical properties of the product were investigated with cyclic voltammetry (CV) measurements carried out by using software controlled conventional three electrode electrochemical cell (CHI660C electrochemical workstation), consisting of the glassy carbon electrode (GCE) was employed as the working electrode (WE), Ag/AgCl as the reference electrode, and the platinum wire as the counter electrode. The working electrode was coated with 0.5 µl of active material ($NiCo_2O_4$) dispersed in 2µl nafion solution. All the electrochemical measurements are performed in a 0.5 M KOH aqueous electrolyte solution at room temperature. The CV measurements were performed at different scan rate of 5 and 30 mVs^{-1}. A potential window in the range from -1.5 to 1.5.7 V was used in this measurement.

RESULT AND DISCUSSION

Structural analysis

Fig.1 shows the X-ray diffraction (XRD) patterns of $NiCo_2O_4$ nanoparticles. The seven diffraction peaks belonging to the JCPDS; 73-1702. All the diffraction peaks can be perfectly indexed to the spinel cubic structure (FCC). The crystalline size of $NiCo_2O_4$ was calculated to be around 15 nm for using the Debye–Scherrer's equation $d = 0.9\lambda/\beta\cos\theta$, where, D is the average crystallite diameter (Å), k is the wavelength of X-ray (1.5406 Å for CuKα radiation), h is the Bragg's angle and β is the full width at half maximum of the synthesized nanoparticles. According to JCPDS, the sample is high purity due to no external peak can be obtained.

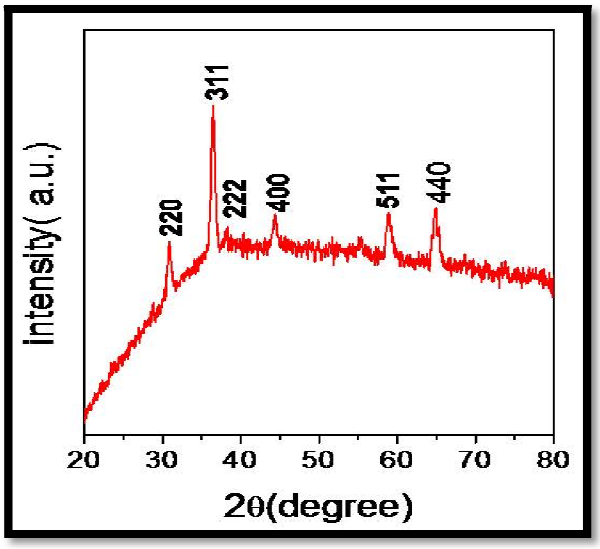


Fig. 1 X-ray diffraction patterns of spinel $NiCo_2O_4$ nanoparticles

Functional group analysis

In order to know the presence of functional groups in the prepared samples, FT-IR spectra of $NiCo_2O_4$ nanoparticles were recorded in the range of 4000-400 cm^{-1} and presented in Fig.2. The absorption bands around at 3626 and 3421 cm^{-1} that could be observed in the sample, which is due to the hydrogen-bonded hydroxyl groups, and several small absorption peaks at around 1000-1600 cm^{-1} are attributed to the bending vibration of O-H bonds. Moreover, presence of two intense bands at 652 cm^{-1} and 530 cm^{-1} were also seen in the spectra of $NiCo_2O_4$. These bands are the feature of a metal–oxygen bond in a spinel-type crystal structure. Two different positions of metal–oxygen bands are due to the difference in bond length between the metal cations and oxygen anions present on tetrahedral and octahedral site [13]

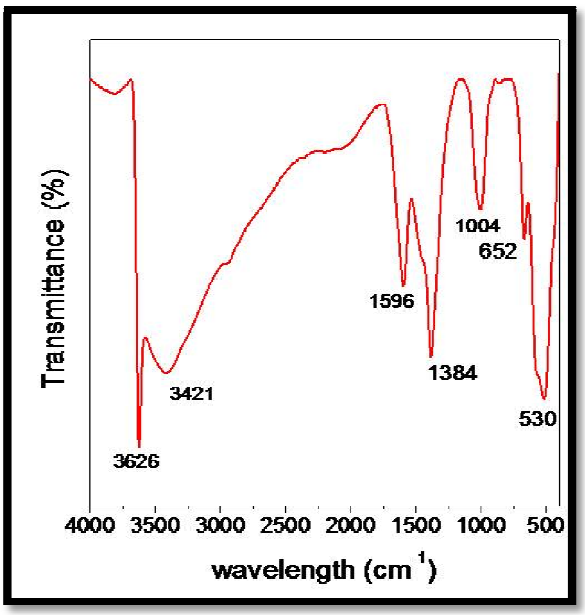


Fig. 2 FTIR spectrum of as-synthesized spinel $NiCo_2O_4$ nanoparticles

Morphological analysis

The morphology and size of the as-prepared $NiCo_2O_4$ nanoparticles were characterized by scanning electron microscopy (SEM). It can be seen from Fig. 3(a) that the spinel nickel cobaltite nanoparticles consists nearly uniform of aggregated spherical shape with diameters of about around 10-15 micrometer. The further observations (Fig 3(b)) show that the big particles are made out of few smallest nanoparticals.

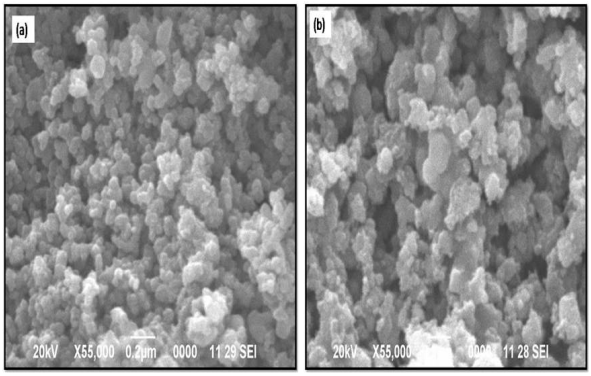
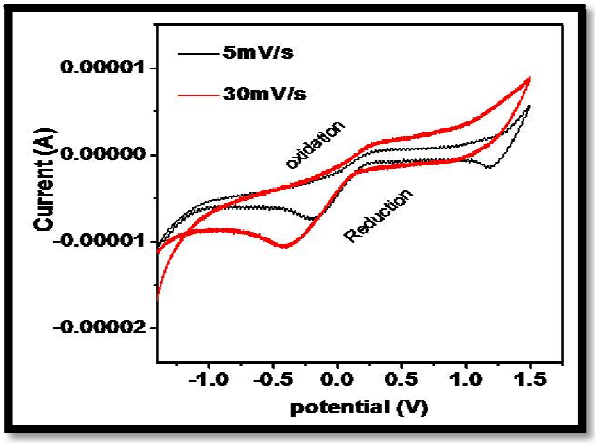


Fig. 3 The FESEM image of the as-synthesized $NiCo_2O_4$ nanoparticles.

Electrochemical studies



The CV measurements on $NiCo_2O_4$ were performed to evaluate the scan rate dependent super capacitive performance of the sample. The Cyclic Voltammetry patterns of the sample are shown in Fig. 4. The CV curves of the sample show perturbed rectangular shape, indicating that the pseudocapacitance nature was entirely different from that of the electric double layer capacitor [14]. The presence of oxidation and reduction peaks indicates a pseudo capacitive behavior of the $NiCo_2O_4$, which is mainly attributed to the Faradaic redox reactions related to M–O/M–O–OH (M represents Ni or Co) associated with OH anions. The redox reactions in the alkaline electrolyte are expressed as follows [15, 16]

$$NiCo_2O_4 + OH^- + H_2O \leftrightarrow NiOOH + 2CoOOH + e^- \quad (2)$$

$$CoOOH + OH^- \leftrightarrow CoO_2 + H_2O + e^- \quad (3)$$

As the scan rate increases, the potential of the anodic and cathodic peaks shift to more positive and negative directions, due to the limitation of the ion diffusion rate to satisfy electronic neutralization during the redox reaction. The specific capacitance values have been calculated from Eq. (4) as

$$Cs = \frac{Q}{\Delta V . m}$$

Where Cs is denoting the specific capacitance, Q the anodic and cathodic charges on each scanning, m is the mass of the electrode material (mg) and Δv is the scanrate (mVs^{-1}). The optimum specific capacitances of binary $NiCo_2O_4$ are 225 Fg^{-1} and 134 Fg^{-1} at 5 and 30 mVs^{-1} respectively. The specific capacitance decreasing with increasing scan rate due to high utilization of the electroactive surface of $NiCo_2O_4$ at low scan rate.

CONCLUSION

The binary spinel $NiCo_2O_4$ nanoparticles were synthesized through co-precipitation method. The XRD results of the $NiCo_2O_4$ are crystalline nature and showed cubic spinel structure. The super capacitive properties of Binary spinel $NiCo_2O_4$ electrodes are investigated using cyclic voltammetry. The electrochemical characteristics showed that the specific capacitance of $NiCo_2O_4$ electrode was achieved a maximum of 225 Fg^{-1} at a scan rate 5 mV/s. The binary spinel nickel cobaltite nanoparticles as one of the most attractive candidate for next generation of supercapacitor device.

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