

# Electrochemical performance of plate-like zinc cobaltite electrode material for supercapacitor applications



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## ABSTRACT

The electrochemical performance of zinc cobaltite-based nanomaterial depends on its shape and morphology. Here we report on the electrochemical performance of plate-like zinc cobaltite nanocrystalline material synthesized via a facile hydrothermal method. The synthesized material was characterized by X-ray diffraction, scanning electron microscopy, energy-dispersive X-ray spectroscopy, X-ray photoelectron spectroscopy, and Brunauer-Emmett-Teller analysis. It was found to be a single-phase zinc cobaltite nanocrystalline material with a cubic spinel crystal structure. The electrochemical performance of the synthesized plate-like zinc cobaltite nanocrystalline material was evaluated by cyclic voltammetry, cyclic chronopotentiometry and electrochemical impedance spectroscopy. The plate-like zinc cobaltite nanocrystalline material displayed a maximum coulombic efficiency of 78% and a maximum specific capacitance of 812 F/g, and it retained 88% of its capacitance after 5100 cycles. Such electrochemical performance may qualify the plate-like zinc cobaltite nanocrystalline material as a potential electroactive material in supercapacitors.

## 1. Introduction

Energy storage materials have recently attracted a great deal of attention. They could be used in many day-to-day applications such as wireless communication devices, hybrid power sources, power tools, fuel cells, and microchips [1,2,3]. Among the various energy storage devices, supercapacitors show great promise in modern electronic devices. This is due to their flexibility, high energy density, high energy conversion rate, long life cycle, simple maintenance, and environmental friendliness [4,5]. On the basis of their charge storage mechanism, supercapacitors are classified as either electric double layer capacitors or pseudocapacitors [6]. Electric double layer capacitors depend on electrostatic charge separation between the electrode-electrolyte interface, whereas pseudocapacitors rely on fast reversible faradaic redox processes [7,8]. Therefore, the physical and electrochemical properties of the electrode material play an important role in the performance of the supercapacitor. Early on, transition metal oxides such as  $\text{MnO}_2$ ,  $\text{NiO}$ , and  $\text{Co}_3\text{O}_4$  and conducting polymers such as polyaniline and polypyrrole were investigated as promising electrode materials for supercapacitor applications [9,10]. They showed promising performance; however, ternary transition metal oxides such as  $\text{MnCo}_2\text{O}_4$ ,  $\text{ZnCo}_2\text{O}_4$ ,  $\text{CoMn}_2\text{O}_4$ , and  $\text{NiCo}_2\text{O}_4$  have attracted more attention because of their higher specific capacitance and richer redox chemistry [11,12,13].

The cobalt-based ternary oxide  $\text{ZnCo}_2\text{O}_4$  has recently attracted attention because of its high conductivity, electroactivity, and excellent electrochemical properties [14,15,16,17,18]. It is also environmentally benign, cost-effective, and abundant [19].  $\text{ZnCo}_2\text{O}_4$  has been shown to be an effective anode material in lithium ion batteries with a high capacity of 900 mA h/g [20]. It has shown potential for supercapacitor applications [21], and it has demonstrated photocatalytic activity [22]. Zinc cobaltite,  $\text{ZnCo}_2\text{O}_4$ , belongs to the spinel crystal structure with the general formula  $\text{AB}_2\text{O}_4$ , where Zn occupies the tetrahedral A sites and Co occupies the octahedral B sites.  $\text{ZnCo}_2\text{O}_4$  is isomorphic to the  $\text{Co}_3\text{O}_4$  spinel crystal structure with the replacement of  $\text{Co}^{2+}$  ions (high spin) by  $\text{Zn}^{2+}$  ions [23].

An electrode material can exhibit efficient mass transfer (i.e., electrolyte penetration and ion transport) if it has a large surface area. This enhances the electrochemical processes and increases the power density and cyclic stability of the supercapacitor [24]. A number of researchers have investigated the electrochemical performance of zinc cobaltite-based material with different morphologies, such as nanoparticles [25], nanowires [26], nanowire arrays [27,28], nanorods [29,30], nanoflakes [20,31], nanotubes [32], hexagonal-like nanostructures [33], urchin-like microspheres [34], core-shell microspheres [35], and porous-structure microspheres [36]. However, it is still a great challenge to develop a simple, economical, environmentally benign, large-scale synthesis method for zinc cobaltite-based electrode material.

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The objective of this work was to study the electrochemical properties and faradaic behavior of plate-like zinc cobaltite as an electrode active material in supercapacitors. We attempted to synthesize plate-like zinc cobaltite nanocrystalline material via a facile hydrothermal method. As far as we know, no such attempt had been undertaken before this work. One of the advantages of the hydrothermal method is the control of the reaction temperature and pressure. The reaction occurs between metallic salts in sodium hydroxide (NaOH) solution without the need for an organic capping agent or a template. The electrochemical properties of the synthesized material were investigated to evaluate the performance and suitability of the plate-like zinc cobaltite material as electrode material in supercapacitors.

## 2. Materials and methods

### 2.1. Chemicals and materials

The starting chemical precursors were zinc nitrate hexahydrate ( $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ ; 98%, Sigma-Aldrich), cobalt(II) nitrate hexahydrate ( $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ ;  $\geq 98\%$ , Sigma-Aldrich), and NaOH pellets (97.5%, SDFCL, Mumbai). The starting chemicals were used as purchased without any further purification. Distilled water was used throughout the hydrothermal synthesis.

### 2.2. Synthesis of plate-like zinc cobaltite material

The starting metal nitrates were weighed in accordance with a molar ratio of Zn to Co of 1:2 and dissolved in 20 ml of distilled water. NaOH was weighed such that the molar ratio of Zn to Na was 1:1 and was dissolved in 10 ml of distilled water. The nitrate solution was stirred until a homogeneous solution was obtained, and then the 10 ml NaOH solution was added dropwise. The resulting solution was subsequently transferred to a Teflon-lined stainless steel autoclave with a capacity of 50 ml. The autoclave was then transferred to a box furnace operating at  $120^\circ\text{C}$  for 12 h. The final solution was allowed to cool to room temperature. The resulting products were removed from the final solution by centrifugation. The products were washed with distilled water seven times and then with acetone three times. The washed products were dried at  $60^\circ\text{C}$  for 8 h. The dried sample was collected and stored for further characterization. A schematic diagram of the hydrothermal synthesis is presented in Fig. 1.

### 2.3. Material characterization

The crystal structure of the synthesized sample was investigated with a Shimadzu LabX XRD-6100 powder X-ray diffractometer. The X-ray diffractometer was operated at 30 kV with a current intensity of 30 mA. The X-ray diffraction (XRD) profile was recorded over the  $2\theta$  range from  $20^\circ$  to  $80^\circ$  with a step size of  $0.02^\circ/\text{min}$  with use of  $\text{Cu K}\alpha$  radiation ( $1.541 \text{ \AA}$ ) at room temperature. The XRD profile was screened against the International Centre for Diffraction Data (ICDD) PDF-2 database. The topology, morphology, and elemental composition of the synthesized sample were examined with a JEOL JSM-6010LA scanning

electron microscope equipped with an energy-dispersive X-ray detector. X-ray photoelectron spectroscopy (XPS) measurements were conducted with a Kratos Analytical Axis Ultra DLD instrument with a monochromatized Al  $\text{K}\alpha_1$  source. X-ray photons of energy 1.486 keV with pass energies of 160 and 40 eV were used for the survey spectrum and the narrow scans, respectively. CasaXPS was used for peak fitting, and the procedure was kept consistent for all the peaks. The surface area was studied by Barrett-Joyner-Halenda algorithm (ASAP 2420 version 2.09) volumetric nitrogen adsorption-desorption experiments.

### 2.4. Electrochemical measurements and working electrode fabrication

Electrochemical properties of the synthesized sample were obtained by cyclic voltammetry (CV), cyclic chronopotentiometry (CP), and electrochemical impedance spectroscopy (EIS). The electrochemical measurements were performed at room temperature with a CHI 7081C electrochemical workstation. CV measurements were collected over a potential range between 0.0 and 0.5 V at various scan rates from 5 to 100 mV/s. The specific capacitance was evaluated from the CP and CV charge-discharge curves. CP measurements were performed at various constant current densities over the potential range from 0 to 0.5 V. Electrochemical impedance spectroscopy measurements were conducted in the frequency region from 1 Hz to 1 MHz. The electrochemical measurements were conducted with a three-compartment cell with a working electrode, platinum wire as the counter electrode, and Ag/AgCl as the reference electrode. The electrodes were immersed in 2 M KOH electrolyte solution during the electrochemical measurements.

The working electrode was fabricated in a fashion similar to that outlined in Ref. [37]. The synthesized sample as an electroactive material was mixed with polyvinylidene fluoride and activated carbon in a weight ratio of 8:1:1. A few drops of 1-methyl-2-pyrrolidinone were then added to the mixture to turn it into a paste. A 0.5 mm thick square nickel plate ( $1 \times 1 \text{ cm}^2$ ) was coated with the paste. The nickel plate was left to dry in air, and then it was annealed at  $80^\circ\text{C}$  for 4 h. The mass of active electrode material was in the range 0.3–0.5 mg. The experimental conditions for the two-electrode system are provided in the supplementary information.

## 3. Results and discussion

### 3.1. Physicochemical properties of the synthesized plate-like zinc cobaltite material

Fig. 2 presents the XRD profile collected from the synthesized material. The diffraction peaks in the XRD profile match quite well with those of the spinel  $\text{ZnCo}_2\text{O}_4$  in accordance with ICDD PDF-2 card no. 00-023-1390. The observed diffraction peaks correspond to the (220), (311), (222), (400), (422), (511), (440), (620), (533), (642), (731), and (751) reflection planes of  $\text{ZnCo}_2\text{O}_4$  with a face-centered cubic spinel crystal structure. No extra diffraction peaks were observed within the limits of the diffractometer, which indicates that the synthesized

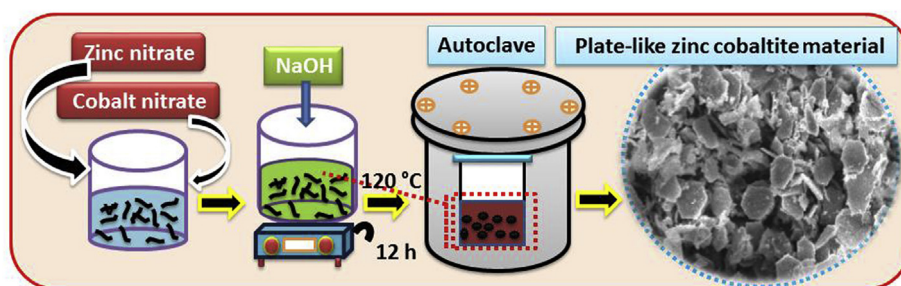


Fig. 1. The facile hydrothermal synthesis method for plate-like zinc cobaltite material.

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