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Hydrothermally Synthesized $CoSn(OH)_6$ Nanoparticles for Electrochemical Performance[*]

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Abstract: Cubic phase $CoSn(OH)_6$ nanoparticles were successfully synthesized by a facile hydrothermal method using NaOH as a mineralizer. The prepared sample was analyzed by using X-ray diffraction (XRD) and the analysis confirmed that the $CoSn(OH)_6$ nanoparticles have a spinel cubic structure with lattice space group Pn3m and mean crystalline size of 25 nm. Field emission scanning electron microscope (FE-SEM) study showed that the cubic with polyhedral shaped surface morphology of the $CoSn(OH)_6$ nanoparticles was seen. The specific capacitance value of 450 Fg^{-1} was calculated at the scan rate of 2 mVs^{-1} as measured by using cyclic voltammetry. In the present investigation, the prepared $CoSn(OH)_6$ nanoparticles are suggested as a potential candidate for supercapacitor applications.

Keywords: Hydrothermal, Nanoparticles, Cubic, Cyclic voltammetry, Specific capacitance.

Introduction

In recent decades, stannate nanostructure materials have attracted special attention because of their tailor-made properties in a wide range of applications in energy storage [1-3]. Among various materials, transition metal oxide-based material play a very important role in supercapacitor applications [4, 5]. However, RuO_2 is considered a good electrode material for supercapacitor because of its high specific capacity of 720 F/g in aqueous acid electrolytes [6]. Nevertheless, the disadvantages are high cost and toxic behavior. Owing to this, the capacitance behavior of different oxide materials such as MnO_2, NiO, Bi_2O_3 and SnO_2 was studied using various electrochemical techniques [7-10]. So, researchers focused on generating alternate materials for supercapacitor applications due to the rising demand and in order to enhance the electrochemical performance of the supercapacitor. Hence, we designed metal stannate hydroxide-based nanomaterials due to their most impressive properties, such as high electron mobility, high power density and long cycle life [11-13]. In this investigation, $CoSn(OH)_6$ nanoparticles are synthesized by using the facile hydrothermal method in the presence of NaOH as a mineralizer to study the structural, morphological and electrochemical performance of the synthesized material.

Experimental Procedure

The chemical reagents ($CoCl_2$. $6H_2O$, $SnCl_4.5H_2O$ and NaOH) were purchased from Merck chemicals (99% pure) and the experiment was carried out without any further purification. $CoSn(OH)_6$ was successfully synthesized by

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using the hydrothermal method [14]. An amount of 30 ml containing 0.06 M of $CoCl_2.6H_2O$ and 0.03 M of $SnCl_4.5H_2O$ was dissolved using deionized water separately to form a transparent solution and then mixed together. An appropriate amount of 3M NaOH solution was added to the mixed solution drop-wise to adjust the pH level up to ~10 under constant stirring. The resultant solution suddenly formed blue color precipitates, which indicates the formation of cobalt stannate hydroxide colloidal suspension. The solution was transferred to a Teflon-coated stainless steel autoclave at 200 °C for 24 h in hot air oven for the hydrothermal reaction. After the hydrothermal reaction, the resultant solution was washed with deionized water and ethanol several times and further dried in an oven at 100 °C for 6 hours. The resultant product was prepared for further characterization.

The synthesized $CoSn(OH)_6$ nanoparticles were characterized by X-ray diffraction (XRD), Field emission scanning electron microscopy (FE-SEM) and Cyclic voltammetry (CV) analyses.

Crystalline structure and size of $CoSn(OH)_6$ were determined by using powder X-ray diffractometer (PAN alytical, Model: PW3040/60 X'pert PRO) with CuK α radiation (λ = 1.54060Å) at 40 kV and 30 mA. The step scan was recorded for 2θ values in the angular range from 10° to 80° with a scanning speed of $10°min^{-1}$. The morphology of the $CoSn(OH)_6$ nanoparticles was observed through the FE-SEM (ZEISS Supra 40VP). Electrochemical performance of the synthesized nanoparticles was measured by cyclic voltammeter (CV) model CHI 660 and the CV measurement was carried out at a potential window between -1.5 V and 2 V.

Results and Discussion

FIG. 1 shows the X-ray diffraction pattern of $CoSn(OH)_6$ nanoparticles. The XRD pattern exhibits strong reflection of hkl planes (111), (200), (220) and (420), indicating that the corresponding 2θ values of 19.3°, 22.8°, 32.5° and 52.2° are indexed for the spinel cubic phase of $CoSn(OH)_6$ from JCPDS card no. 74-0365. The average crystal size was determined by Debye Scherer formula [15].

$$D = \frac{K\lambda}{\beta Cos\theta}; \qquad (1)$$

where D is the crystallite size, K (= 0.89) is a constant related to the shape of the crystal, λ is the wavelength of the radiation employed, β is the full width at half maximum (FWHM) of the obtained reflection peak in radians and θ is the Bragg diffraction angle. The calculated mean crystalline size was found to be 25 nm. The XRD study confirmed that the as prepared sample was cubic structured $CoSn(OH)_6$ nanoparticles without any impurities and with primitive lattice Pn3m space group.

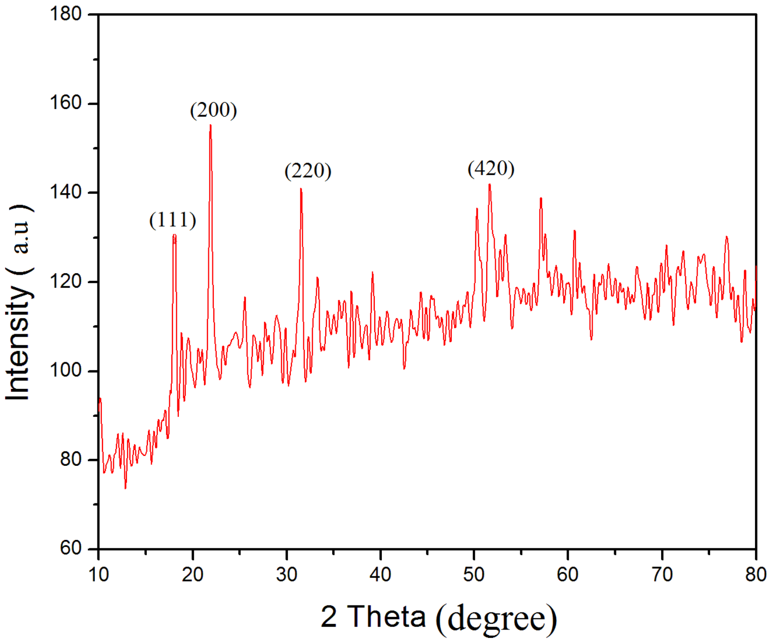

FIG. 1. XRD pattern of $CoSn(OH)_6$ nanoparticles.

The morphology of $CoSn(OH)_6$ nanoparticles was observed by FE-SEM micrograph measurements. FIG. 2 shows the FE-SEM image of $CoSn(OH)_6$ nanoparticles. It clearly demonstrates that the nanoparticles are aggregated in polyhedral and cubic shape [16]. From the figure, it can be seen that the $CoSn(OH)_6$ nanoparticles are evenly distributed in the range of 20-50 nm, which indicates that the FESEM result of $CoSn(OH)_6$ nanoparticles closely matches the XRD result.

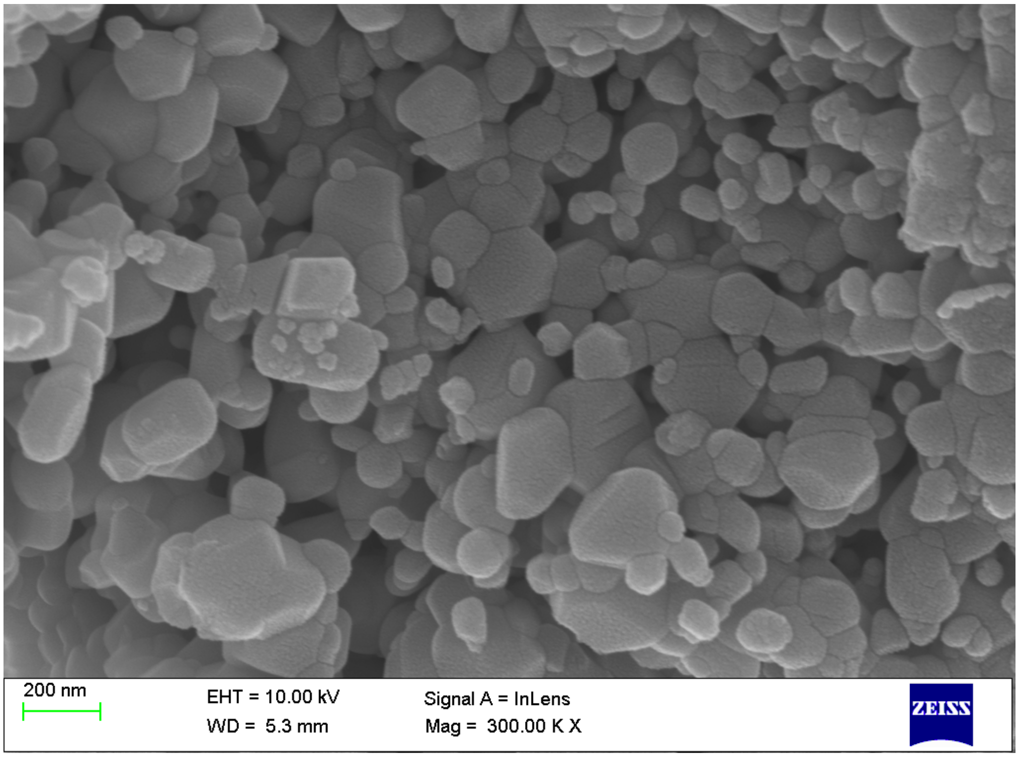

FIG. 2. FE-SEM image of CoSn(OH)6 nanoparticles.

For investigating electrochemical performance, the CV analysis was carried out in the potential window between -1.5 V and 2 V at different scan rates as shown in FIG. 3. The $CoSn(OH)_6$ nanoparticles were tested as an electrode to evaluate the electrochemical performance in a three-electrode system [17]. It exhibits quasi rectangular shape of CV curve with scan rate values from 2 to 50 mVs^{-1}. The specific capacitance of synthesized $CoSn(OH)_6$ nanoparticles can be calculated using the following formula [18]:

$$Cs = \frac{Q}{\Delta v.m} \quad (2)$$

where Cs is the specific capacitance, Q is the anodic charge, m is the mass of the electrode material (mg) and Δv is the scan rate ($mV\ s^{-1}$). While increasing the scan rate, the shape of curve reduction indicates that the change in the specific capacitance value gets decreased from 450 Fg^{-1} at 2 mVs^{-1} to 23 Fg^{-1} at 50 mVs^{-1} as shown in Table 1. The CV curve exhibits a shape different from the identical rectangular shape, which indicates the pseudo capacitance nature of the $CoSn(OH)_6$ nanoparticles [19]. In addition, it confirms the changes in specific capacitance while increasing scan rate owing to the pseudo capacitance nature of $CoSn(OH)_6$ nanoparticles. It indicates that at lower scan rates, ionic diffusion takes place and electrons interact with the inner and outer surfaces of the nanoparticles due to Faradaic reaction [16]. At increased scan rates, ionic diffusion takes place only at the outer surface of the nanoparticles [20]. The $CoSn(OH)_6$ nanoparticles have a high specific capacitance value of 450 Fg^{-1} at a scan rate of 2 mVs^{-1}. It can be concluded that $CoSn(OH)_6$ nanoparticles are suitable for supercapacitor applications.

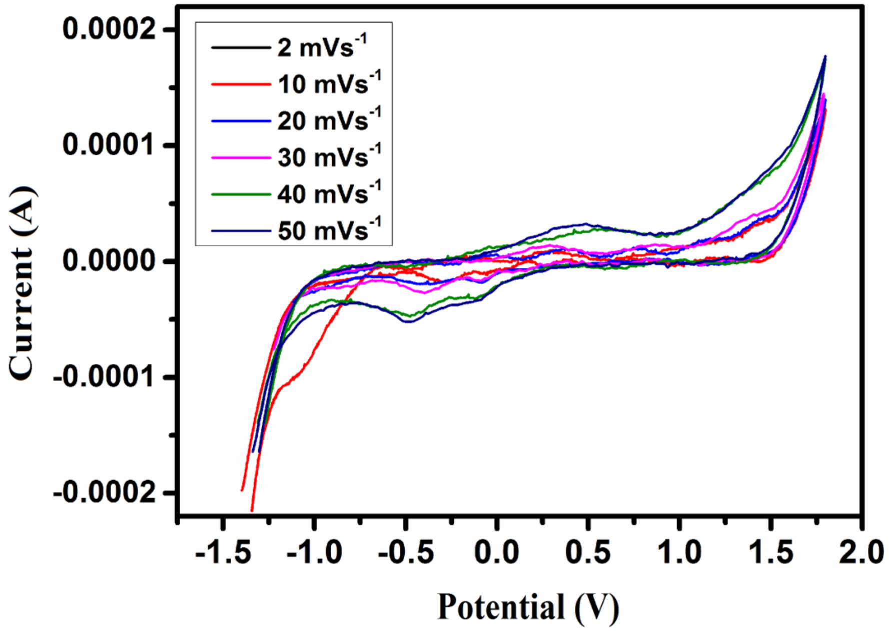

FIG. 3. Cyclic voltammetry curves of CoSn(OH)6 nanoparticles at different scan rates.

TABLE 1. Specific capacitance of CoSn(OH)$_6$ nanoparticles at different scan rates.

CoSn(OH)$_6$ nanoparticles						
Scan rate (mVs^{-1})	2	10	20	30	40	50
Specific capacitance (Fg^{-1})	450	136	65	44	32	23

Conclusions

CoSn(OH)$_6$ nanoparticles were synthesized successfully by using the facile hydrothermal method. The XRD analysis confirmed cubic structure of CoSn(OH)$_6$ and a mean crystal size of 25 nm. FE-SEM reveals the synthesized product to be in polyhedral with agglomerated cubic shape. The average crystalline size from XRD matches well the FE-SEM result. The superior specific capacitance value of 450 Fg^{-1} was obtained at the scanning rate of 2 mVs^{-1}. We deem that this facile process and specific capacitance performance enlighten the CoSn(OH)$_6$ nanoparticles to be a potential candidate for supercapacitor applications.

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