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Synthesis of multi metal sulfide based electrode material for high performance supercapacitor

K S Ch Srinivasa Rao *, V R P Parameswar G, Mahita Chekuri and Kalyani Chalapaka

Department of Physics, Sir C R Reddy College (A), Eluru, Andhra Pradesh-534006, India.

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Abstract

Transition metal sulfides are referred to as good materials for effective energy storage with high electrochemical performance due to their multi oxidation states. Herein, we prepare cobalt-molybdenum-sulfide nano composite denoted as CMS through hydrothermal method. The CMS@NF exhibit great electrochemical performance of specific capacity 112.8 mAh/g and 60 % of capacity retention at 10 A/g current density after 10,000 cycles. These results indicate that CMS@NF is a promising positive electrode material in the field of energy storage technology.

Keywords: CMS@NF; Hydrothermal method; Specific capacity; Supercapacitor

1. Introduction

The growing demand for health-monitoring devices, smart wearable watches, and related applications has made high-performance energy storage devices (ESDs) that are flexible, lightweight, and environmentally sustainable critically important [1–4]. Supercapacitors can be categorized into two types—electrical double-layer capacitors (EDLCs) and pseudocapacitors (PCs)—based on their electrode materials, charge storage mechanisms, and operational principles [5]. The electrode material significantly influences the performance of supercapacitors (SCs), as it directly affects parameters such as specific capacitance, energy density, power density, and cycle life. Therefore, the development of high-performance electrode materials is critical for optimizing the electrochemical properties and overall efficiency of SCs [6–10]. The energy storage efficiency of supercapacitors is mainly affected by several crucial factors, such as the electrochemical characteristics of the electrode materials, the selection of electrolyte, and the working potential range. Extensive research has been focused on creating and improving advanced electrode materials with optimal structural characteristics to boost electron movement and ion diffusion effectiveness. Transition metal sulfides (TMSs) exhibit both electric double-layer charge adsorption/desorption and faradaic redox reactions, contributing to enhanced energy density. Cobalt-based sulfides are notable electrode materials among transition metal sulfides (TMSs) because of their excellent conductivity, diverse redox states, layered configuration, adjustable stoichiometry, straightforward synthesis into different structures, and remarkable theoretical capacity. Wang et al. synthesized novel silver fungus-like CoS (SFC) via solvent thermal method, which delivers high specific capacity of 48.67 mAh/g at 1 A/g current density [11]. In particular, multi metallic sulfides offer more faradaic reaction sites compared to binary metallic sulfides.

Herein, we synthesized multi metallic sulfide such as cobalt-molybdenum-sulfide (CMS) through hydrothermal method. CMS electrodes show significantly high specific capacity of 112.8 mAh/g at 1 A/g current density and retain 60 % of its capacity after 10,000 cycles in 6M KOH aqueous electrolyte solution (3 - electrode configuration).

* Corresponding author: K S Ch Srinivasa Rao

2. Material and method

2.1. Materials

Cobalt (II) nitrate hexahydrate (Co $(NO_3)_2 \cdot 6H_2O$), thiourea (CH_4N_2S), sodium molybdate dihydrate ($Na_2MoO_4.2H_2O$), activated carbon, polyvinylidene fluoride (PVDF), N-Methyl-2-pyrrolidone (NMP) and all the chemical reagents are all analytically pure without further purification.

2.2. Synthesis of CMS composite

0.43g of Co $(NO_3)_2 \cdot 6H_2O$, 0.362g $Na_2MoO_4 \bullet 2H_2O$ and 0.7612g of thiourea were respectively dissolved in 60 mL DI water under vigorous stirring for 1 h to obtain a translucent solution. The mixture is then transferred into 100 mL autoclave and heated to 170°C for 10 h. Finally, washed with ethanol, DI water three times, and dried at 70°C for 8 hours.

2.3. Characterization techniques

The surface morphology of the sample was analysed by the using field-emission scanning electron microscopy (Joel JIB 4700F FIB-SEM).

3. Results and discussion

Fig. 1 (a-d) show the surface morphogical images of as prepared electrode material CMS composite. All images show a porous cubical morphology with a smooth surface and this porous structure leads to enhance the transportation of electrolyte through electrode material.

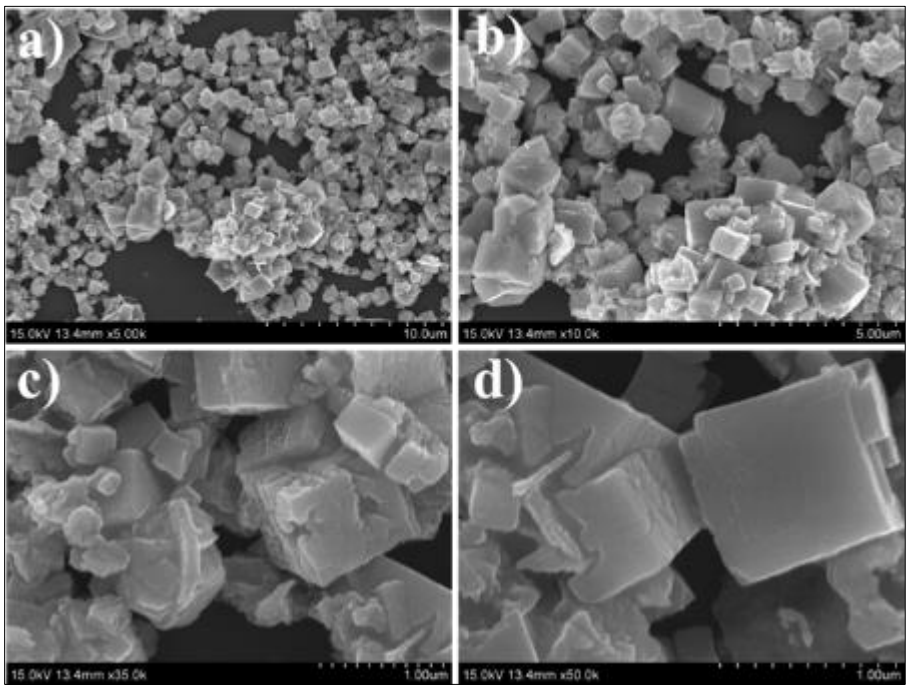

Figure 1 (a -d) FESEM images of CMS electrode material with different resolutions

Electrochemical tests were performed using a three-electrode setup connected to a Biologic SP 150e electrochemical workstation in 6 M KOH solution at room temperature. The as-prepared sample (CMS@Ni foam), Ag/AgCl, and platinum wire served as the working, reference, and counter electrodes, respectively. Cyclic voltammetry (CV) measurements were conducted over a potential range of 0 – 0.5 V (vs. Ag/AgCl). Electrochemical impedance spectroscopy (EIS) was carried out at open-circuit potential, covering frequencies from 0.01 Hz to 100 kHz. Chronopotentiometry (CP) measurements were recorded within a potential window of 0 – 0.4 V (vs. Ag/AgCl). The specific capacity (C, mAh/g) was determined using the following equation [12] (Eq. 1):

$$C_s = \frac{I \times \Delta t}{3.6 \times m} \ldots\ldots\ldots\ldots\ldots (1)$$

Here, C_s, I, and m represent the specific capacity (mAh/g), discharge current (A), and mass loading of the active material (g), respectively. Δt denotes the discharge time (s).

Fig. 2(a) shows the CV curves of as prepared electrode material CMS@NF at various scan rates 5, 10, 20, 40, 70, 100 mV/s with a voltage range 0 to 0.5 V. All the CV (cyclic voltammetry) curves exhibit visible redox peaks, indicating that it possesses good electrochemical performance compared to binary sulfides. All CV curves present a pair of redox peaks, and the oxidation peak shifts moderately from 0.35–0.4 V with increasing the scan rate from 5 to 100 mV/s. The CP (chronopotentiometry) curves CMS@NF are approximately symmetrical and shows apparent plateau region at charge/discharge process, which suggest Faradaic reactions (Fig. 2(b)). The CMS@NF electrode material achieves maximum specific capacity 112.8 mAh/g at 1 A/g current density. The calculated specific capacitances are 78.2, 65.9, 59.4, 54.8 and 42.9 mAh/g with scan rates 2, 3, 4, 5 and 10 A/g. The electrochemical impedance spectroscopy (EIS) was done to know about the charge transfer resistance (R_{ct}) of electrode material. Fig. 2(c) shows the electrochemical impedance spectra (EIS) of CMS@NF, where at the high frequency there is a small semicircle in which intercepts the real axis at 0.65 Ω, showing the small series resistance and the excellent charge transport during electrochemical processes. The rate capability at different current densities is shown in the Fig. 2(d), the capacity of CMS@NF is gradually decreases with increase in current density.

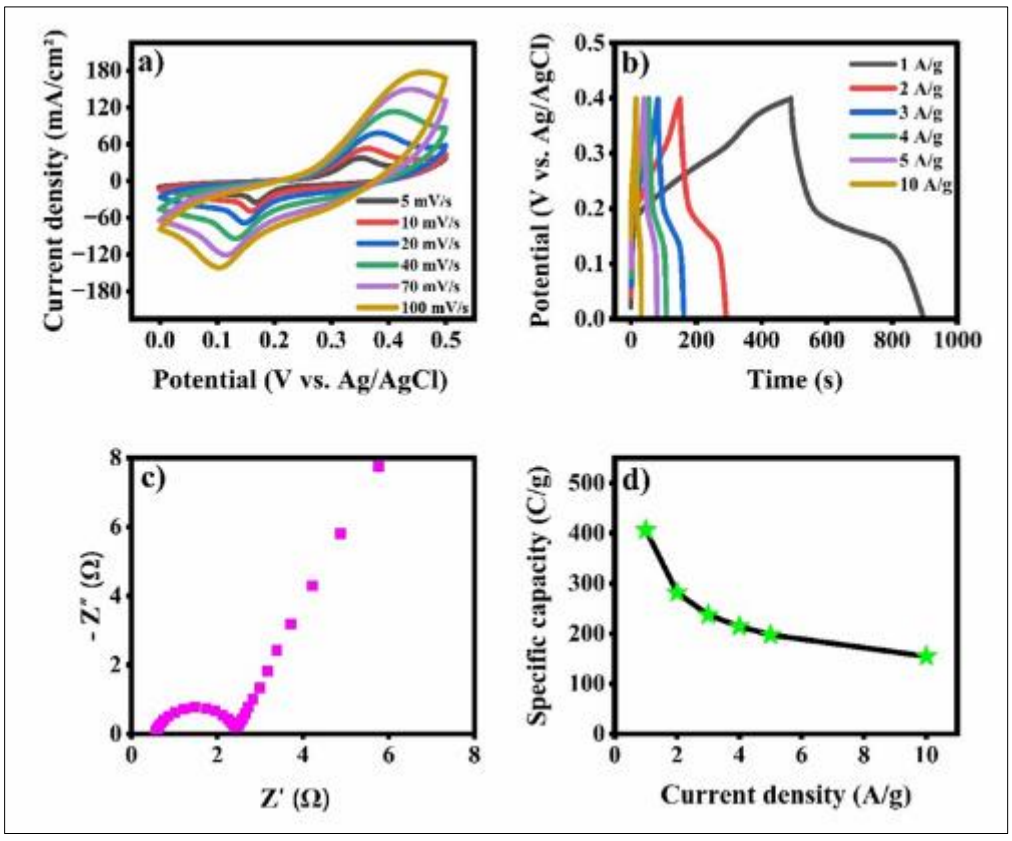

Figure 2 Electrochemical performance of CMS@NF a) CV curves at different scan rates; b) CP curves at different current densities; c) EIS plot; & d) Rate capability at different densities

In general, the slope of log (scan rate, mV/s) vs log (peak current, mA) graph is used to determine the b-value of electrode material. Determining the value of b helps identify the dominant charge storage mechanism in an electrochemical system. A diffusion-controlled process is thought to be the charge storage mechanism if b = 0.5, while a surface controlled capacitive process is thought to be the performance if b = 1. A mixed mechanism involving both surface-controlled and diffusion-controlled contributions if 0.5 < b < 1.

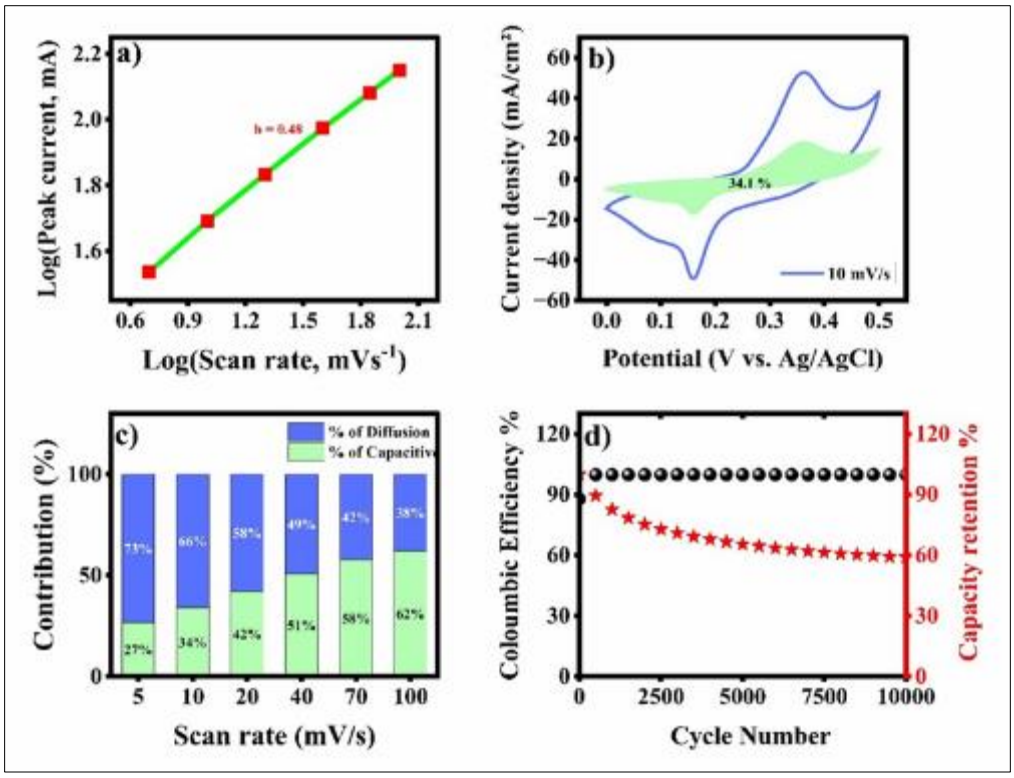

Figure 3 Electrochemical performance of CMS@NF a) log (scan rate) Vs log (peak current) of CNMS-MXene sample; b) Capacitive contribution at 10 mV/s; c) Capacitive contribution ratios at different scan rates; & d) Cyclic stability

The slope of the log (scan rate, mV/s) vs log (peak current, mA) plot shown in the Fig. 3(a) was determined to be 0.58. This value clearly shown that the energy storage in the supercapacitor took place via two different processes viz., capacitive process and Faradaic process. As illustrated in Fig. 3(b), at scan rate of 10 mV/s, the capacitive contributed 34.1 % while the diffusion contributed 65.9 % for CMS@NF electrode. The comparative charge storage contributions at 5, 10, 20, 40, 70 and 100 mV/s scan rates as shown in the Fig. 3(c). The repeating charge/discharge processes at the current density of 10 A/g in Fig. 3(d) show the good capacity retention about 60% and high coulombic efficiency > 90% even after 10,000 cycles, which indicates the good charge transfer ability after long-term cycling. The findings indicated that the CMS@NF electrode material exhibited a battery like energy storage mechanism [13].

4. Conclusion

CMS@NF electrode material was successfully prepared via hydrothermal approach. The electrochemical measurements of CMS@NF were analysed in 6M KOH aqueous electrolyte. It exhibited excellent electrochemical performance with specific capacity of 112.8 mAh/g at 1 A/g along with ~60% capacity retention at 10 A/g after 10,000 cycles.

Compliance with ethical standards

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Disclosure of conflict of interest

The authors declare no conflict of interest.

Authors contribution

K S Ch Srinivasa Rao: Writing – original draft, Formal analysis. V R P Parameswar G: Visualisation and validation. Mahitha Chekuri: Resources. Kalyani Chalapaka: Writing – review & editing, Resources.

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