



Original scientific paper

Scalable synthesis of FeCo_2O_4 nanostructured electrodes on flexible stainless-steel mesh *via* chemical bath deposition for supercapacitor applications

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Abstract

FeCo_2O_4 thin films were successfully deposited onto stainless steel mesh (SSM) substrates using a simple, scalable, and binder-free chemical bath deposition method. To the best of our knowledge, this is the first report demonstrating FeCo_2O_4 thin films directly deposited on SSM as binder-free electrodes for supercapacitor applications. The conductive SSM promoted strong adhesion and efficient charge transport, while the nanostructured FeCo_2O_4 morphology enhanced electrolyte-ion diffusion. The FeCo_2O_4 film deposited for 3 hours exhibited a higher specific capacitance of 739 F g^{-1} at 1 mA cm^{-2} , compared to 522 F g^{-1} of the film formed for 2 hours under identical conditions. The corresponding energy densities were calculated as 23 and 16 Wh kg^{-1} , respectively, confirming the improved electrochemical performance with increased deposition time. These results demonstrate that optimized deposition time significantly enhances the charge-storage performance of FeCo_2O_4 /SSM electrodes, highlighting their potential for high-performance supercapacitor applications.

Keywords: Pseudocapacitive energy storage; chemically deposited thin films; binary transition metal oxide; metal mesh; binder-free electrode.

Introduction

The rapid growth in global energy demand, coupled with the continued dependence on fossil fuels, has intensified concerns regarding greenhouse gas emissions, environmental degradation, and climate change [1]. The transition toward renewable energy technologies, including solar and

wind power, offers a promising pathway toward sustainable energy generation. Nevertheless, the intrinsically intermittent and fluctuating nature of these renewable sources necessitates efficient energy-storage systems capable of rapidly storing and delivering energy with high efficiency and long-term stability [2]. Consequently, the development of advanced electrochemical energy-storage technologies with high power density, enhanced energy density, rapid charge-discharge capability, and prolonged cycle life has become an important research priority [3].

Among the available energy-storage technologies, supercapacitors have attracted considerable attention because of their high-power density, fast charge-discharge kinetics, excellent rate capability, and long operational lifetime [4]. Their electrochemical energy-storage mechanism, which combines electrical double-layer capacitance and/or rapid surface or near-surface redox reactions, enables substantially faster energy delivery than conventional batteries. However, the relatively low energy density of supercapacitors remains a critical limitation for their broader implementation in applications requiring both high power and increased energy storage [5]. According to the relationship between energy density and capacitance, increasing the electrochemically accessible surface area, enhancing reversible redox activity, and improving charge and ion transport within the electrode are essential strategies for overcoming this limitation. Therefore, the rational design of highly active electrode materials together with appropriate electrode architectures and scalable fabrication routes is essential for advancing the practical performance of supercapacitors.

Nanostructured transition-metal oxides have emerged as attractive electrode materials because of their multiple oxidation states, abundant electrochemically active sites, tenable morphology, and high theoretical charge-storage capacity [6]. In particular, binary transition-metal oxides have received increasing interest because the incorporation of two metal cations can generate synergistic redox chemistry and provide more favourable electronic and electrochemical characteristics than several corresponding single-metal oxides. Among them, iron cobalt oxide (FeCo_2O_4) is considered a promising candidate for pseudocapacitive energy storage owing to the coexistence of multiple oxidation states of Fe and Co, which can participate in reversible faradaic reactions [7-9]. The coupled redox activity of Fe and Co ions, together with the structural and compositional versatility of the spinel oxide framework, can provide abundant active sites and facilitate efficient charge-storage reactions. However, the electrochemical performance of FeCo_2O_4 strongly depends on its crystallinity, morphology, particle size, active surface area, electrode architecture, and electrical contact with the current collector. Thus, controlling the growth of FeCo_2O_4 into an accessible nanostructured architecture remains important for maximizing its practical electrochemical response.

Beyond the intrinsic properties of the active material, the current collector plays a critical role in determining the overall performance and mechanical integrity of a supercapacitor electrode. Conventional substrates such as nickel foam, carbon cloth, carbon-based frameworks, and copper foams provide conductive pathways and interconnected porous structures; however, their relatively high cost, material-specific limitations, or inadequate mechanical characteristics can restrict their suitability for economical and large-area electrode fabrication [10-12]. Stainless steel mesh (SSM), in contrast, offers a combination of low cost, electrical conductivity, mechanical robustness, corrosion resistance, and a flexible interconnected architecture, making it an attractive alternative substrate for binder-free electrodes [13,14]. Its open porous structure can facilitate electrolyte penetration and provide a three-dimensional conductive network for electron transport. More importantly, the direct growth of active materials onto SSM can establish intimate electrical contact and minimize the use of polymeric binders and conductive additives, which may otherwise introduce electrically resistive interfaces and inactive mass.

The deposition strategy is equally important because it governs the nucleation, growth, morphology, adhesion, and interfacial characteristics of the active material. A variety of synthesis techniques, including chemical vapour deposition, solvothermal processing, hydrothermal synthesis, electrodeposition, and related wet-chemical routes, have been employed for preparing transition-metal oxide electrodes [15-17]. Although these methods can provide good control over material structure, several approaches may involve elevated temperatures or pressures, prolonged reaction durations, multiple processing steps, or relatively sophisticated instrumentation. Such requirements can increase energy consumption and processing costs and may limit straightforward scale-up. Chemical bath deposition (CBD) provides an appealing alternative because it is a simple, low-temperature, solution-based technique that can facilitate the direct formation of adherent thin films on substrates with relatively uncomplicated experimental infrastructure [18]. The ability to deposit functional materials over large-area substrates, combined with low equipment requirements and potentially reduced processing costs, makes CBD particularly attractive for the scalable fabrication of electrochemical electrodes.

Despite the considerable interest in FeCo_2O_4 -based supercapacitor electrodes, a significant research opportunity remains in integrating the intrinsic redox activity of FeCo_2O_4 , the conductive porous architecture of SSM, and the simplicity of direct CBD growth within a single electrode design. In particular, many reported electrode architectures rely on binders, additional conductive agents, or multistep synthesis processes, which can increase fabrication complexity and introduce additional interfaces between the active material and current collector. Moreover, achieving uniform and strongly adhered FeCo_2O_4 nanostructures directly on a flexible, low-cost, conductive mesh while maintaining efficient electrolyte accessibility and electron transport remains a relevant challenge. Therefore, a facile deposition strategy capable of simultaneously addressing material-substrate adhesion, electrical connectivity, electrolyte accessibility, and fabrication scalability is highly desirable.

In this work, FeCo_2O_4 thin films were directly deposited for different durations onto stainless steel mesh (SSM) using a facile and scalable CBD route to develop a binder-free electrode architecture for supercapacitor applications. The SSM substrate was employed as both a conductive current collector and a porous structural framework for the nucleation and growth of FeCo_2O_4 , thereby eliminating the need for polymeric binders and additional conductive additives. The structural and morphological characterisation of the deposited FeCo_2O_4 films was systematically investigated, with particular emphasis on the development of a nanostructured surface favourable for electrolyte interaction. The electrochemical behaviour of the FeCo_2O_4 /SSM electrode was subsequently evaluated to elucidate its charge-storage characteristics and the contribution of the Fe- and Co-based redox processes. The study aims to establish the relationship between the directly grown FeCo_2O_4 architecture and its electrochemical response while demonstrating the potential of CBD-derived FeCo_2O_4 /SSM electrodes as a low-cost and scalable platform for advanced supercapacitor applications.

Experimental

Experimental resources and reagents

All analytical-grade reagents employed in this study were procured from Sigma-Aldrich and used without further purification. All aqueous solutions were prepared using double-distilled water to minimize the influence of ionic and other impurities on the synthesis process.

Analysis techniques

The crystal structure and phase composition of the FeCo₂O₄ thin films were investigated using an X-ray diffractometer (Bruker D8) equipped with a Cu K α_1 radiation source. The surface morphology, film architecture, and microstructural features of the FeCo₂O₄ thin films were examined using a scanning electron microscope (SEM; Hitachi, Japan). The electrochemical experiments were carried out on the CHI 608E electrochemical analyser instrument at room temperature.

Electrochemical measurements

Electrochemical experiments were carried out at room temperature in 1 M KOH aqueous electrolyte unless otherwise mentioned. Cyclic voltammetry (CV) and galvanostatic charge-discharge (GCD) were carried out in a standard three-electrode system with platinum wire and Ag/AgCl electrode as the counter and reference electrodes, respectively. Cyclic voltammetry profiles were recorded at scan rates ranging from 5 to 100 mV s⁻¹ within a potential window of 0.0 to 0.5 V (vs. Ag/AgCl). The GCD profiles were obtained at current values of 1, 3, 5 and 7 A (current densities of 1, 3, 5 and 7 A cm⁻²) within a potential window of 0.0 to 0.5 V.

The specific capacitance (C_{sp} / F g⁻¹) is calculated according to Equation (1)

$$C_{sp} = \frac{I \Delta t}{m \Delta V} \quad (1)$$

where I / A, Δt / s, m / g and ΔV / V represent the applied current, discharge time, the mass of the electroactive material and potential window, respectively. The mass loading of FeCo₂O₄ deposited on the SSM substrate was determined from the difference between the masses of the substrate before and after deposition. The measured active-material mass loadings were 0.79 mg for a thin film of FeCo₂O₄ formed through 2 h (FCO-2h) and 1.0 mg for a thin film of FeCo₂O₄ formed through 3 h (FCO-3h). These respective mass loadings were used to calculate the specific capacitance of the electrodes. The FeCo₂O₄/SSM electrodes were prepared with a geometric area of 1 cm², and GCD measurements were performed at a current of 1 mA, corresponding to the current density of 1 mA cm⁻².

Specific energy density and specific power density are the most important parameters that determine the energy storage capability of the supercapacitor electrode in a supercapacitor device. The energy density (E / Wh kg⁻¹) and power density (P / W kg⁻¹) values of a single electrode were calculated using Equations (2) and (3) [19]:

$$E = \frac{0.5 C_{sp} \Delta V^2}{3.6} \quad (2)$$

$$P = \frac{E 3600}{\Delta t} \quad (3)$$

where ΔV is the potential window and Δt is the discharge time.

Synthesis of FeCo₂O₄ thin film on stainless-steel mesh

CBD method was used for deposition of thin FeCo₂O₄ films on SSM. The SSM substrates were initially cleaned ultrasonically in acetone, 10 % HCl solution, and deionized water for 20 min in each medium to remove organic contaminants, surface oxides, and residual impurities. For the deposition process, a precursor solution was prepared by dissolving 0.1 M Fe(NO₃)₃·9H₂O and 0.2 M Co(NO₃)₂·6H₂O in 50 mL of double-distilled water under continuous magnetic stirring for 2 h to obtain a homogeneous solution. The pH of the resulting solution was subsequently adjusted to approximately 11.0±0.5 by the dropwise addition of NH₄OH under constant stirring.

The prepared chemical bath was maintained at 60 °C, and the pre-cleaned SSM substrates were vertically immersed in the solution for either 2 or 3 hours. Under these conditions, the controlled

release of hydroxide ions promoted the nucleation and growth of the Fe-Co hydroxide precursor directly on the SSM surface through the chemical bath deposition process. Following deposition, the coated substrates were thoroughly rinsed with deionized water to remove loosely adhered species and subsequently dried. The deposited Fe-Co hydroxide films were then annealed at 300 °C for 1 h using a controlled heating rate of 2 °C min⁻¹, resulting in the formation of crystalline FeCo₂O₄ thin films [20,21].

Results and discussion

Structural and morphological analysis

The crystal structure of the synthesized FeCo₂O₄ (FCO) thin films deposited for 2 h and 3 h on stainless steel mesh (SSM) was investigated using X-ray diffraction (XRD), as shown in Figure 1. Both diffraction patterns exhibited characteristic reflections corresponding to the cubic spinel structure of FeCo₂O₄. The observed diffraction peaks at $2\theta \approx 18.3, 30.1, 35.4, 37.0, 43.1, 53.5, 57.0$ and 61.6° were indexed to the (111), (220), (311), (222), (400), (422), (511) and (440) planes, respectively. These reflections are well indexed to the cubic spinel structure of FeCo₂O₄ according to JCPDS No. 22-1086. No additional impurity-related peaks were observed, confirming the phase purity of deposited FCO films. The additional peaks marked with (*) are attributed to SSM substrate. The obtained diffraction patterns are in good agreement with previously reported FeCo₂O₄ synthesized using different methods [22,23].

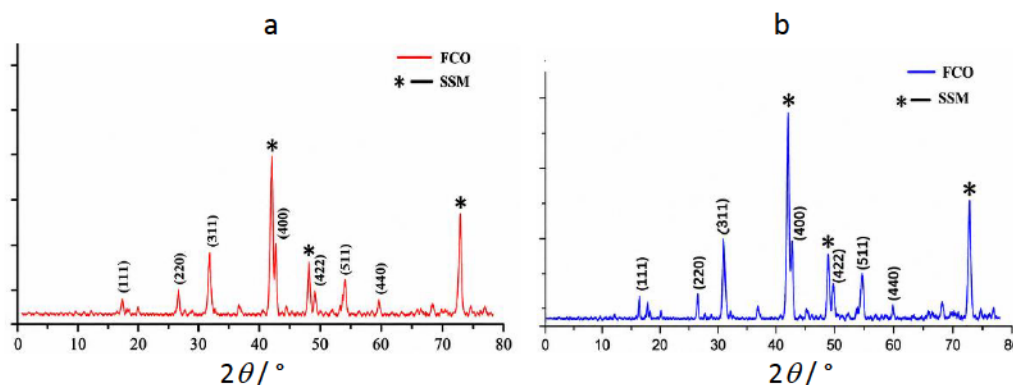


Figure 1. XRD pattern of a) FCO-2h and b) FCO-3h films deposited on SSM

The average crystallite size of the FCO films was estimated using the Debye-Scherrer Equation (4). The calculation was based on the most intense (311) diffraction peak at $2\theta \approx 35.4^\circ$. The calculated average crystallite size was found to be 42.6 nm for FCO-2h and 39.2 nm for FCO-3h, respectively.

The slight decrease in crystallite size with increasing deposition time indicates the formation of relatively finer crystallites during prolonged deposition.

$$D = \frac{k\lambda}{\beta \cos \theta} \quad (4)$$

In Equation (4), D is the average crystallite size, k is the Scherrer constant (taken as 0.89), λ is the X-ray radiation wavelength (0.15406 nm), β is the full width at half maximum (FWHM) of the diffraction line, and θ is the Bragg's diffraction angle in radians [24].

To gain insight into the morphology and possible growth mechanism of the FeCo₂O₄ (FCO) thin films, scanning electron microscopy (SEM) was employed. The formation of the deposited thin film can be broadly described in terms of successive stages involving nucleation, aggregation, coalescence, and subsequent growth of the nanostructured features. Figure 2(a-d) presents SEM

micrographs of the FCO-2h and FCO-3h films deposited directly onto the SSM substrate at different magnification. At $200\times$ magnification the FCO-2 electrode shows relatively sparse and non-uniform deposition on the SSM, whereas the FCO-3 electrode exhibits denser and more uniform coverage of the mesh fibres. At $5000\times$ magnification the FCO-2 electrode displays relatively isolated agglomerated particles, while the FCO-3 h electrode shows a higher density of fine, heterogeneous nano-structured particles and rougher surface morphology. The increased deposition with longer deposition time provides greater surface coverage and potentially more electrochemically accessible active sites [25].

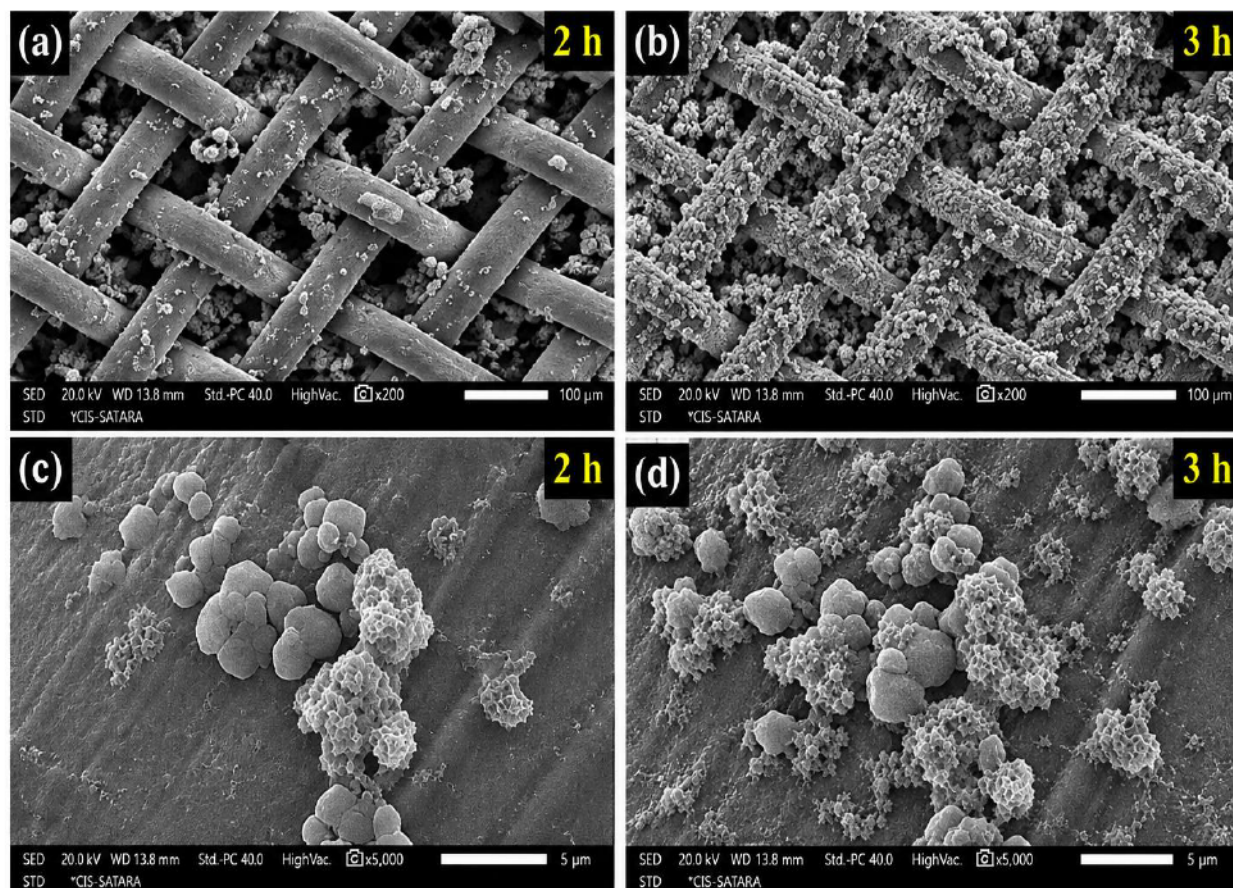


Figure 2. SEM images of FCO films deposited on SSM for (a, b) 2 h and (c, d) 3 h at different magnifications

To investigate the influence of CBD deposition time on the electrochemical performance, FCO-2h and FCO-3h electrodes were comparatively evaluated using CV. As shown in Figure 3a and b, both electrodes exhibited an increase in current response with increasing scan rate, accompanied by an enlargement of the area enclosed by the CV curves, indicating enhanced electrochemical activity and charge-storage kinetics. Notably, at identical scan rates, the FCO-3h electrode exhibited a significantly higher current response and a larger enclosed CV area than the FCO-2h electrode. This observation suggests that the 3 h deposited film possesses superior electrochemical charge-storage capability. The enhanced performance of FCO-3h may be attributed to the higher loading of electroactive FeCo_2O_4 material and the increased availability of electrochemically active sites resulting from the longer deposition time. Figure 3(c) presents the CV curves of the FCO-2h and FCO-3h electrodes at a scan rate of 5 mV s^{-1} . The variation in the integrated CV area with deposition time provides an indication of the charge-storage behaviour of the electrodes [26].

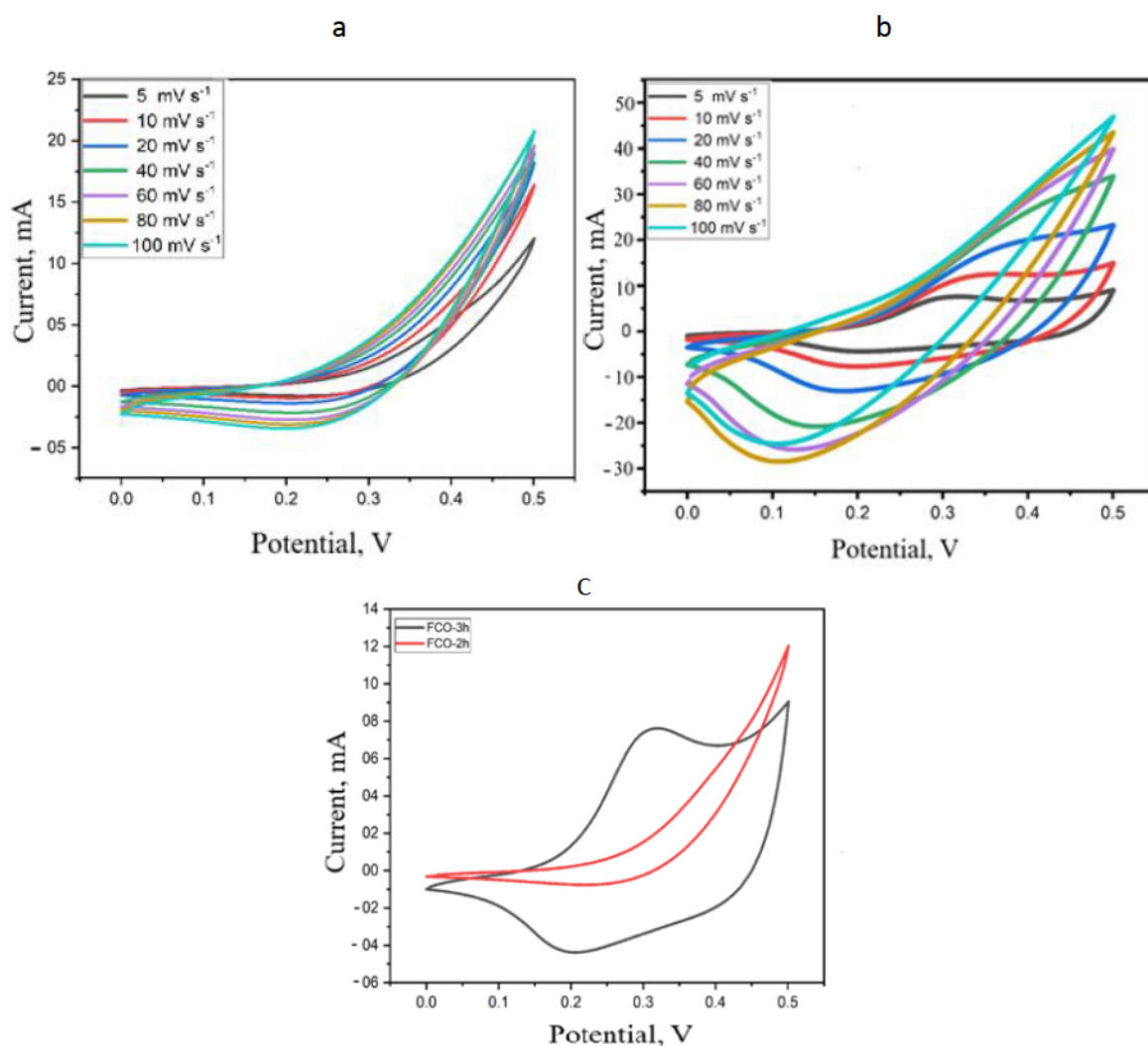


Figure 3. Cyclic voltammetry (CV) curves of the FCO@SSM electrodes at different scan rates (5 to 100 mV s^{-1}) for (a) FCO-2h and (b) FCO-3 h, and (c) comparative CV curves (5 mV s^{-1}) of FCO-2 h and FCO-3h

To understand the capacitive nature of the FCO@SSM electrodes, galvanostatic charge-discharge (GCD) measurements were carried out. The nonlinear nature of the discharge curves suggests the involvement of faradaic redox processes, confirming the pseudocapacitive behaviour of the FeCo_2O_4 electrode.

GCD performances of the FCO-2h and FCO-3h electrodes were evaluated at different current densities of 1, 3, 5 and 7 mA cm^{-2} ($I = 1, 3, 5$ and 7 A) (Figure 4a and 4b). The 2 h deposited electrode delivered lower specific capacitances of 522, 346, 323 and 308 F g^{-1} at the corresponding current values. Figure 4b for the FCO-3h electrode exhibited higher specific capacitance values of 739, 652, 555 and 456 F g^{-1} at currents of 1, 3, 5 and 7 mA, respectively. The superior electrochemical performance of the 3 h deposited electrode can be attributed to the increased amount of electroactive material and improved development of the active electrode surface during the longer deposition time, which provides more accessible redox-active sites for charge storage [3]. Although the specific capacitance decreased with increasing current density for both electrodes, the FCO-3h electrode maintained comparatively higher capacitance throughout the investigated current-density range.

These results demonstrate that the deposition time significantly influences the electrochemical charge-storage behaviour of the FCO@SSM electrode, with the 3 h deposition condition providing enhanced capacitive performance compared with the 2 h deposited electrode [19]. The specific capacitance decreases with increasing current density for both electrodes, which can be attributed to the reduced ion-diffusion time and incomplete utilization of the electroactive sites at higher

current densities. The 3 h deposited electrode exhibits a higher specific capacitance than the 2 h electrode over the investigated current-density range, indicating better electrochemical utilization and charge-storage capability [27].

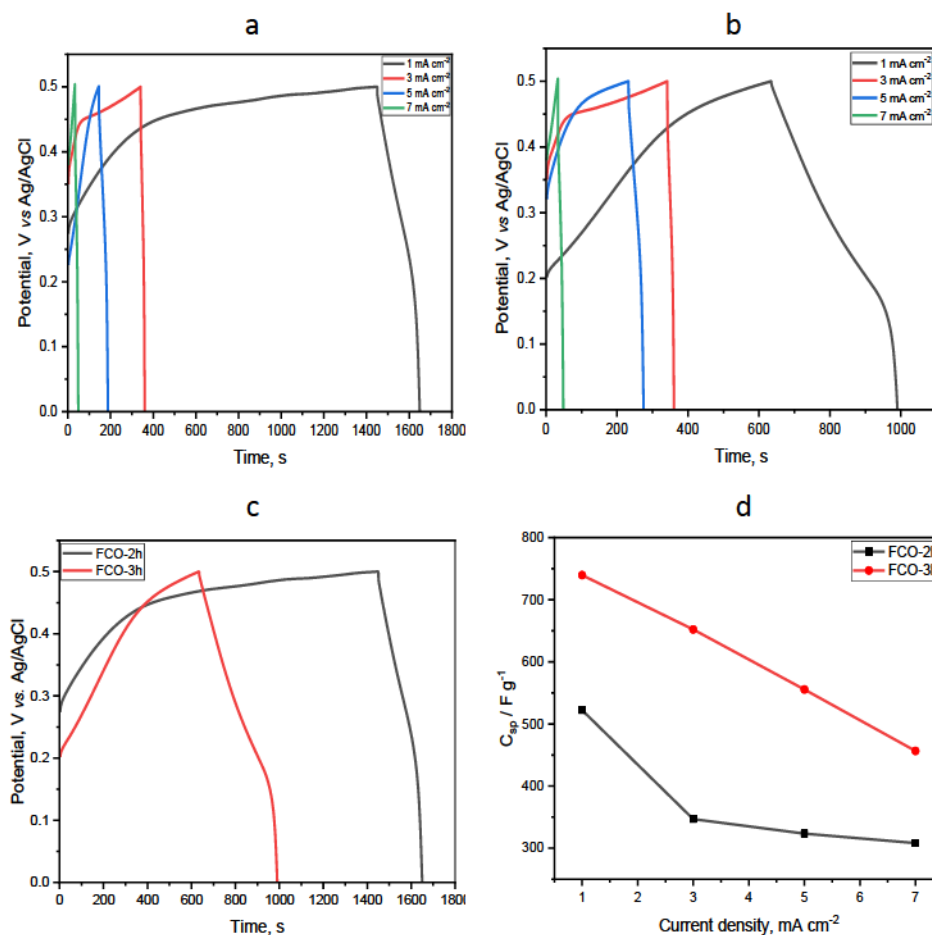


Figure 4. Galvanostatic charge-discharge curves of FCO@SSM electrodes: recorded at different current densities for (a) FCO-2h, (b) FCO-3h, (c) comparative GCD curves of FCO-2h and FCO-3h recorded at a current density of 1 mA cm^{-2} and (d) variation of specific capacitance with current density for FCO-2h and FCO-3h electrodes

Specific energy and power density values for both FCO electrodes were calculated using Equations (2) and (3) and C_{sp} values of 522 F and 739 F g^{-1} obtained at a current density of 1 mA cm^{-2} . For FCO-2h and FCO-3h, the resulting energy densities were 16 and 23 Wh kg^{-1} , while power density values were 240 and 303 W kg^{-1} , respectively. These results indicate that the longer deposition time would be more favourable for enhanced energy storage than the 2 h electrode.

Comparative analysis

The comparative literature analysis shows that FeCo_2O_4 electrodes exhibit promising supercapacitor performance depending on the synthesis method and substrate used (Table 1). Reported specific capacitance values range from 260 to $\sim 1963 \text{ F g}^{-1}$, with hydrothermal synthesis generally providing high capacitance. In comparison, the sponge-like FeCo_2O_4 electrode synthesized by the simple and low-cost CBD method on stainless-steel mesh (SSM) in this work delivers specific capacitance of FCO-3h 739 F g^{-1} and FCO-2h 522 F g^{-1} at 1 mA cm^{-2} . Although its capacitance is lower than some hydrothermally synthesized electrodes, the use of CBD and flexible SSM offers advantages of simplicity, scalability, low cost and binder-free electrode fabrication, making the present approach attractive for practical supercapacitor applications.

Table 1. Comparative study of the FeCo_2O_4 @SSM formed by CBD with previously reported FeCo_2O_4 electrode material

Electrode material	Method of preparation	Substrate	Specific capacitance	Ref.
FeCo_2O_4	Hydrothermal	Flexible SSM	1963 F g^{-1} at 1 A g^{-1}	[7]
FeCo_2O_4	Wet-chemical co-precipitation	Cu (flexible & stiff)	1048.07 to 1690.14 F g^{-1}	[24]
FeCo_2O_4	Reflux condensation synthesis	Flexible SSM	260 F g^{-1} at 1 mA cm^{-2}	[28]
FeCo_2O_4	Hydrothermal method	Nickel foam	679.94 F g^{-1} at 1 A g^{-1}	[29]
FeCo_2O_4	Hydrothermal synthesis	Nickel foam	$247.54 \text{ mAh g}^{-1}$ at 2 mA cm^{-2}	[30]
FeCo_2O_4	Hydrothermal method	Powder	393.5 F g^{-1} at 1 A g^{-1}	[31]
FeCo_2O_4 -2 h	Chemical bath deposition	Flexible SSM	522 F g^{-1} at 1 mA cm^{-2}	This work
FeCo_2O_4 -3 h			739 F g^{-1} at 1 mA cm^{-2}	

Conclusion

In conclusion, FeCo_2O_4 thin films were directly deposited onto stainless steel mesh by a facile chemical bath deposition method and binder-free electrodes were investigated for supercapacitor applications. XRD analysis confirmed the formation of crystalline FeCo_2O_4 with the characteristic diffraction peaks corresponding to its spinel structure. SEM analysis revealed increased FeCo_2O_4 deposition with a rougher and more porous nanostructured surface for the 3 h electrode compared with the relatively compact morphology observed at 2 h. The binder-free FCO-3h single electrode exhibited superior electrochemical performance, delivering 739 F g^{-1} at 1 mA cm^{-2} , compared with 522 F g^{-1} for FCO-2h. The FCO-3h single electrode could achieve an energy density of 23 Wh kg^{-1} , compared with 16 Wh kg^{-1} for FCO-2h, while the calculated power densities were 240 and 303 W kg^{-1} , respectively. The enhanced performance of FCO-3h is attributed to its favourable nanostructured morphology, efficient electrolyte accessibility, and synergistic redox activity of Fe and Co species. Overall, the FeCo_2O_4 /SSM architecture combined with the CBD technique offers a simple, economical, and effective strategy for developing binder-free electrodes for supercapacitor applications.

Author contributions: PSG - conceptualization, methodology, investigation, experimental work, data curation, formal analysis and writing - original draft; PST: - supervision, project administration, writing - review and editing and corresponding author; SHM - formal analysis, data validation, supervision, and writing - review and editing.

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