

Recent Advances in Hydrothermal Synthesis of MFe_2O_4 Spinel Ferrite Nanostructures for High-Performance Supercapacitor Applications: A Comprehensive Review

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ABSTRACT: This review systematically compiles two decades of research on the hydrothermal (HT)/solvothermal (ST) synthesis of MFe_2O_4 and its derivatives for supercapacitor (SC) applications. It examines the influence of synthesis parameters on electrode composition, morphology, and electrochemical performance, including specific capacitance, energy/power density, cycling stability, and the roles of dopants, surfactants, electrolytes, and electrode architectures. HT methods enable precise control over nanostructure morphology (nanospheres, nanosheets, nanorods, etc.), which significantly improves ion diffusion pathways and active surface area. Composite electrodes incorporating graphene, rGO, CNTs, or activated carbon routinely achieve specific capacitances $>2000 \text{ F/g}$ and energy densities $>70 \text{ Wh/kg}$. Doping with Zn, Ni, Co, Mn, or rare-earth elements (e.g., La) enhances redox activity, electrical conductivity, and structural stability, often yielding $>90\text{--}98\%$ capacitance retention after 10,000 cycles. Surfactants (CTAB, PVP, SDS) effectively regulate particle size, prevent agglomeration, and increase specific surface area. Key characterization tools XRD, SEM/TEM, XPS, BET, and electrochemical techniques (CV, GCD, EIS) provide critical correlations between structure, composition, and performance. The review establishes performance benchmarks, highlights optimal synthesis structure property relationships, and offers actionable guidelines for designing scalable, high-performance, long-lifespan MFe_2O_4 based SC electrodes through sustainable HT routes.

KEYWORDS: Hydrothermal synthesis, MFe_2O_4 ferrites, SCs, Nanostructured electrodes, Electrochemical performance.

I. INTRODUCTION

Research on the HT synthesis of CuFe_2O_4 (CFO) for SC applications has emerged as a critical area of inquiry due to the increasing demand for efficient, sustainable energy storage systems. SCs offer high power density, rapid charge-discharge rates, and long cycle life, making them promising alternatives to conventional batteries [1-2]. Over the past decade, advancements in transition metal oxides, particularly spinel ferrites like CFO, have demonstrated enhanced electrochemical properties owing to their multiple redox states and environmental friendliness [3]. The HT method has gained prominence for synthesizing nanostructured CFO with controlled morphology and high surface area, which are crucial for improving SC performance [4]. The practical significance is underscored by reports of specific capacitances exceeding 2000 F/g and energy densities above 40 Wh/kg in CFO based composites [5]. Despite these advances, challenges remain in optimizing the composition, morphology, and electrochemical stability of CFO electrodes synthesized hydrothermally. Several studies highlight a knowledge gap in understanding the influence of dopants, surfactants, and electrolyte types on the electrochemical performance and cycling stability of CFO based materials [6]. Contrasting reports exist regarding the effectiveness of graphene-based composites versus pure CFO or other metal oxide hybrids, with some indicating superior capacitance and stability in composites [7], while others emphasize the cost-effectiveness and simplicity of pure CFO synthesis [8-9]. The lack of consensus on optimal synthesis parameters and electrode formulations limits the practical deployment of these materials [10]. Addressing this gap is essential to enhance energy density and power density without compromising long-term cycling stability [11]. The conceptual framework for this review integrates the synthesis of CFO via HT methods, the role of electrode material composition and morphology,

and their impact on electrochemical performance metrics such as specific capacitance, energy density, power density, and cycling stability [1,3-4]. The interplay between dopants, surfactants, and electrolytes is examined to elucidate mechanisms underlying performance enhancement [12]. In contemporary times, HT technology has established its relevance across numerous disciplines within the realms of science and technology, encompassing a diverse array of fields, including materials science, earth science, metallurgy, physics, chemistry, and biology, among others (Figure 1). In light of the extensive applicability and the escalating significance of the HT methodology, it has undergone substantial enhancements throughout a process characterized by ongoing development.

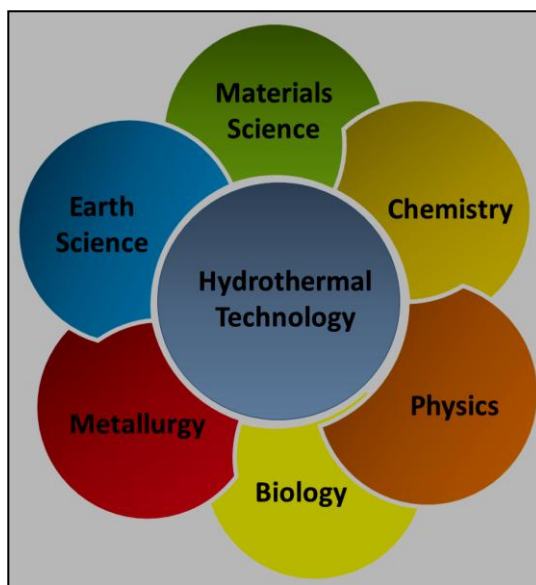


Figure 1. Domain of application for HT methodologies¹³.

II. RELATED WORK

This framework guides the systematic evaluation of characterization techniques and electrochemical analyses to inform material design strategies.

III. SIGNIFICANCE OF THE SYSTEM

This comprehensive review systematically assesses the advancements in the HT synthesis of CFO for SC electrodes, focusing on factors such as composition, morphology, performance, and stability. It elucidates the influences of dopants, surfactants, and electrolytes, thereby addressing existing discrepancies and facilitating the informed design of high-performance, resilient CFO based materials.

IV. METHODOLOGY

Hydrothermal Method

The designation "hydrothermal" has its roots in the field of geology, emerging in the mid-19th century when geologists endeavoured to replicate HT conditions in order to investigate the genesis of specific minerals and rocks. Consequently, HT techniques began to be utilized for the growth of single crystals; the methodology for powder preparation has been refined over the course of nearly two centuries. The HT technique is defined as the utilization of an aqueous solution as a reaction medium within a specialized closed reaction apparatus (Figure 2), wherein a high-temperature and high-pressure reaction milieu is established through the application of heat and pressure (or via the vapor pressure generated from the system itself). This procedure facilitates the dissolution and subsequent recrystallization of a substance that exhibits low solubility or is entirely insoluble under standard conditions. The overarching procedural steps associated with the HT technique are illustrated in Figure 2.

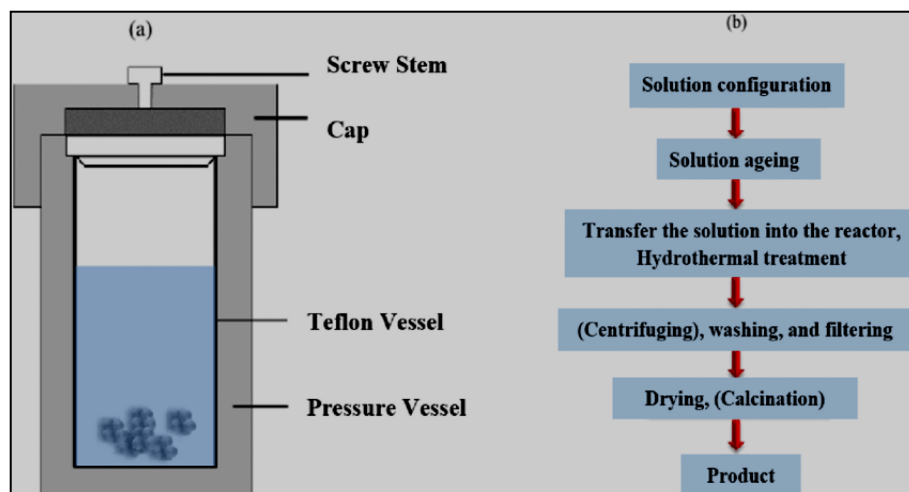


Figure 2. Hydrothermal synthesis: (a) a schematic representation of a conventional apparatus, and (b) the standard procedures involved in the process¹³.

V. HYDROTHERMAL SYNTHESIS OF SPINEL FERRITE FOR SC APPLICATION

This section delineates the academic landscape pertaining to the hydrothermally synthesized CFO utilized in SCs, concentrating on aspects such as composition, morphology, electrochemical efficiency, stability, doping agents, surfactants, electrolytic mediums, and methodologies for characterization (Table 1). It conducts a comparative analysis of synthesis methodologies, emphasizes the tuning of morphological and compositional attributes, and establishes performance benchmarks to elucidate the manner in which synthesis parameters and material modifications enhance the properties of CFO based electrodes.

Synthesis Parameters

Recent studies predominantly employed HT and ST techniques, adjusting temperature, reaction time, and precursor ratios to tailor CFO morphology and phase purity [4,11,14]. Structural and electrochemical enhancements were often achieved through dopants such as Zn, Ni, and La or by forming composites with graphene and other carbon materials [15,19-20]. Surfactants like PVP, gelatin, and organic capping agents were used to control particle size and dispersion, improving morphology and surface area [8]. Additionally, several works explored mineral-based or green synthesis routes to promote sustainability and efficient resource utilization [11,14,21].

Electrochemical Performance

Reported electrochemical performance varies considerably across studies, with specific capacitance ranging from approximately 145 F/g to over 2100 F/g, particularly high in graphene-based composites and doped CFO materials [3-5]. Energy densities span from about 5 Wh/kg to more than 70 Wh/kg, often improved through hybrid configurations and core-shell architectures [15-16]. Power densities also show notable enhancement when conductive additives and optimized morphologies are employed, reaching several kW/kg in some reports [15].

Cycling Stability

Cycling stability across the literature is generally robust, with most CFO-based electrodes retaining over 80% of their capacitance after 1,000–10,000 cycles, reflecting good long-term durability [4,6,16]. Enhanced stability is consistently observed in composites incorporating carbon materials or dopants, which outperform pure CFO electrodes [3,7]. Notably, several studies reported retention values exceeding 90%, underscoring the effectiveness of advanced synthesis strategies and tailored material designs [5,22].

Dopant and Surfactant Effectiveness

Dopants such as Zn, Ni, La, and Cu substitution improved redox activity, charge transfer, and structural stability [15,19-20,23]. Surfactants and capping agents-controlled particle growth and morphology, leading to enhanced surface area and electrochemical performance [8,12]. Carbon-based additives like graphene, rGO, and carbon spheres significantly increased conductivity and cycling life [3-4,15].

Characterization Techniques

Across the reviewed literature, XRD was universally used to verify phase purity and crystallinity of synthesized CFO materials. Morphological characterization relied heavily on SEM and TEM, which provided insights into particle size,

shape, and composite distribution [4,7,24]. Electrochemical evaluation commonly employed cyclic voltammetry, galvanostatic charge–discharge, and electrochemical impedance spectroscopy to assess capacitance, rate capability, and resistance characteristics [4,6,25]. Additionally, several studies incorporated advanced techniques such as XPS, Raman spectroscopy, BET surface area analysis, and magnetic measurements to further elucidate surface chemistry, structural interactions, and magnetic behaviour [5,12,26].

Table 1. Comprehensive Summary Table delineating various spinel ferrites alongside their corresponding characterization methodologies.

HT synthesis	Electrochemical Performance	Cycling Stability (capacitance retention (%) after number of charge–discharge cycles)	Dopant and Surfactant Effectiveness	Characterization Techniques	Study
CuFe ₂ O ₄ /CuO@rGO nanocomposite with asymmetric cell	Specific capacitance >2110 F/g; energy density 40.44 Wh/kg; power density 780 W/kg	93% @ 10,000 cycle	rGO enhances conductivity and stability	XRD, SEM, TEM, electrochemical tests (CV, GCD)	[4]
N- doped graphene oxide with CuFe ₂ O ₄	Specific capacitance 348 F/g; energy density 35.79 Wh/kg; power density 883 W/kg	87% @ 2000 cycles	N-doping improves electron/ion transport	XRD, SEM, TEM, electrochemical analysis	[6]
chalcopyrite ore on Ni foam; cauliflower morphology	Areal capacitance 525 mF/cm ² ; energy density 8.9 mWh/cm ² ; power 233 mW/cm ²	109% @ 1350 cycles	Ore-based synthesis without dopants	XRD, FTIR, XPS, SEM, TEM	[14]
CuFe ₂ O ₄ nanofiber with 3D graphene sheet composite	Specific capacitance 489 F/g; high power density	95% @ 10,000 cycles	3D graphene enhances surface area and stability	XRD, SEM, TEM, CV, GCD, EIS	[7]
Bimetallic CuFe ₂ O ₄ ternary hybrid composites with polyaniline and acetylene black	Specific capacitance 279.48 F/g; energy density 38.81 Wh/kg; power 125 W/kg	85% @ 6000 cycles	Polyaniline and acetylene black improve conductivity	CV, GCD, EIS	[10]
Co-precipitation of CuFe ₂ O ₄ with ZnO nanoparticle doping	Specific capacity 409.7 C/g; energy density 17.8 Wh/kg; power density 3125 W/kg	Not specified	ZnO doping enhances electrochemical activity	CV, GCD	[15]
CuFe ₂ O ₄ /carbon microspheres hybrid	Specific capacity 320 C/g; energy density 40.9 Wh/kg; power density 11248 W/kg	82.4% @ 5000 cycles	Carbon spheres improve surface area and conductivity	XRD, CV, GCD	[15]
CuFe ₂ O ₄ –NiFe ₂ O ₄ core-shell nanostructures	Specific capacity 1366 C/g; energy density 72 Wh/kg	97% @ 10,000 cycles	Core-shell structure enhances ion diffusion	XRD, Raman, BET, SEM, TEM	[16]
CoFe ₂ O ₄ nanoflake arrays on Ni foam	Areal capacity 9.7 C/cm ² ; energy density 6.01 Wh/m ²	89.7% @ 5000 cycles	Low Cu doping in synthesis solution	SEM, XRD, XPS, BET, CV, GCD, EIS	[17]
Ternary Ni _x Fe _y Cu _z (CO ₃)(OH) ₂ electrode	Specific capacity 271.8 mAh/g; energy density 55.1 Wh/kg	84.3% @ 10,000 cycles	Ternary metal compounds improve ion transport	XRD, BET, electrochemical tests	[18]

VI. THEORETICAL AND PRACTICAL IMPLICATIONS

Theoretical insights from recent studies indicate that HT synthesis enables precise morphology control of CFO such as hollow spheres, nanospheres, and nanofibers thereby enhancing ion diffusion and pseudocapacitive behaviour [7]. Composites with rGO or NGO improve conductivity and charge-transfer kinetics, supporting theories that hybrid structures overcome the intrinsic limitations of metal oxides [6]. Dopant incorporation (Zn, Ni, La) further tunes electronic structure and redox activity, improving pseudocapacitive response and stability [15]. Magnetic effects in CFO-based materials also influence charge storage, aligning with emerging concepts of magnetically ordered pseudocapacitors [12]. Additionally, electrolyte composition and surfactants significantly affect ion transport and interfacial resistance, reaffirming the importance of electrolyte optimization [26]. Overall, observed pseudocapacitive and battery-like behaviors support hybrid charge-storage models in spinel ferrites.

Recent studies highlight significant practical implications of hydrothermally synthesized CFO-based materials for commercial SCs, demonstrating high capacitance, energy density, and durability, along with cost-effective and scalable production [4,15]. Incorporation of rGO and graphene enhances conductivity and mechanical stability, supporting applications in flexible and wearable electronics [7]. Green and mineral-based synthesis routes further indicate environmentally sustainable pathways for electrode fabrication [11]. Tunability through dopants and composite engineering enables targeted performance optimization for grid storage, electric vehicles, and consumer devices [27]. Asymmetric device configurations pairing CFO with activated carbon show promising practical architectures [6]. Additionally, the magnetic and multifunctional nature of CFO composites broadens their relevance to catalysis and sensing applications, offering cross-sector technological potential.

VII. FUTURE RESEARCH DIRECTIONS

Future research should prioritize systematic optimization and reporting of HT synthesis parameters, including surfactant type, reaction time, and temperature, to ensure reproducibility. Extended high-current cycling tests, advanced electrochemical and magnetic analyses, and detailed investigation of dopant effects and defect structures are needed. In situ/operando techniques should be applied to monitor structural evolution, while scalable green synthesis, mass-loading effects, and standardized testing protocols will further support practical advancement of CFO electrodes.

VIII. CONCLUSION AND FUTURE WORK

The literature on HT synthesis of spinel ferrite for SC applications shows that synthesis parameters strongly influence morphology, composition, and electrochemical behavior. HT and ST routes enable controlled fabrication of diverse nanostructures, while adjustments in precursor ratios, temperature, time, and the use of surfactants such as PVP or gelatin refine particle size, porosity, and dispersion. Dopants including Zn, Ni, and La, as well as composites with graphene, carbon materials, or conductive polymers, significantly enhance conductivity, redox activity, and stability, yielding specific capacitances frequently above 800 F/g and occasionally over 2000 F/g. Electrolytes such as KOH or Na₂SO₄ further modulate charge storage mechanisms. Characterization methods—XRD, SEM, TEM, and electrochemical techniques provide structural and performance insights, though inconsistencies in testing limit direct comparisons. Despite good cycling stability, long-term durability, scalability, and mechanistic understanding remain challenges. Continued improvement in synthesis control and standardized evaluation is crucial for practical device integration.

Conflicts of Interest:

The authors declare no conflict of interest.

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