

Spinel Ferrite-Based Electrode Materials for Supercapacitors: A Review

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Abstract: Transition metal ferrites (MFe_2O_4 , $M = Co, Ni, Mn, Zn, Mg, Cu$) and their composites are a very promising family of pseudocapacitive electrode materials for supercapacitors and asymmetric supercapacitors (ASCs) because of their multi-electron redox activity, wide operating potential windows up to 1.8 V, chemical stability and low cost. The crystallite size, surface area and morphology progress are vital in synthesis routes such as hydrothermal/solvothermal processing, binder-free electrodeposition, co-precipitation and sol-gel auto-combustion. Hybridization of ferrites with conducting polymers and conductive carbon matrices and direct growth on 3D conductive substrates are effective ways to overcome the intrinsic limitations such as low electrical conductivity and structural degradation during cycling. This review provides a real-world guide to the material design, key performance indicators and the standardization requirements for high-energy and high-power next-generation energy storage devices.

Keywords: Spinel ferrite nanostructures; Supercapacitors; Sol-gel synthesis; Electrodeposition; Binder-free electrodes; Morphological engineering.

I. INTRODUCTION

With the world's growing demand for energy, the importance of developing energy storage components that are efficient and environmentally friendly is becoming ever more significant. Supercapacitors (SCs) play an important role in the scaling of renewable energy as they offer a smooth transition between batteries, fuel cells and traditional capacitors in terms of their capacity and power delivery [1]. SCs have emerged as a premier energy storage technology driven by the demands of high-power density and ultrafast charge-discharge cycles. SCs combine battery-like capacity with capacitor-like speed in a lightweight, environmentally friendly footprint. The electrochemical behaviour of the electrode, the electrolyte used and the limits of the working potential are the main factors affecting the efficiency of SCs [2–4]. SCs use reversible ion adsorption at the electrode-electrolyte interface to store and release energy. Generally, they are divided into electric double layer capacitors (EDLCs), pseudocapacitors and hybrid supercapacitors. EDLCs are devices that store charge electrostatically using high surface area carbon electrodes, such as activated carbon, carbon aerogels, porous carbon, carbon nanotubes (CNTs), and graphene. The limited specific capacitance of conventional carbon EDLCs has driven recent research toward advanced materials such as dual-functionalized carbon architectures[5]. Recent progress on inexpensive carbon precursors demonstrates their great potential for scalable energy storage. Specifically, the carbons derived from coal can be upgraded to highly graphitic structures by flash Joule heating (FJH), which intensely increases the electrical conductivity and overall electrochemical performance[6]. Physical electrostatic storage offers fast charge delivery but suffers from intrinsic limitations in specific capacitance (C_{sp}). Pseudocapacitive electrode materials such as transition metal oxides (RuO_2 , MnO_2 , CoO_2) and conducting polymers like polyaniline (PANI) and polypyrrole (PPy) store charge by fast faradaic processes like surface redox and intercalation reactions in comparison. These mechanisms allow pseudocapacitors to achieve significantly higher



specific capacitance and energy density than conservative EDLCs [7–11]. Carbon nanotubes (CNTs), possessing excellent mechanical resilience, high electrical conductivity, large surface area and tunable functionality, have emerged as very promising building blocks for flexible energy storage [12]. Thus, MXene/graphene hydrogels emphasize the necessity of robust, conductive matrices in the design of high-performance energy storage systems. However, the sheet restacking of graphene-based materials is a common problem that affects their electrochemical performance, in spite of the excellent electrical conductivity, large surface areas, and porous architectures. Holey graphene (HG) nanosheets are introduced to effectively suppress interlayer aggregation to promote rapid ion transport and maintain the accessible surface area [13]. Due to their mechanical flexibility, stretchability and biocompatibility, wearable and skin-mountable micro-supercapacitors have attracted considerable interest for on-body electronics. Moreover, they exhibit excellent electrochemical performance with high-rate capability and long-term cycling stability [14]. Thus, metal oxides are good candidates for electrodes of supercapacitors. Metal ferrites (MFe_2O_4 , $\text{M}=\text{Co, Ni, Mn, Fe, or Cu}$) and their composites have shown promising potential in different energy storage systems such as supercapacitors and lithium-ion batteries (LIBs) [15–19], SCs [20–26]. Metal ferrites and their composites are highly promising for asymmetric supercapacitor (ASC) applications due to some unique advantages such as (i) capability of multiple multi-electron redox reactions; (ii) higher working potential window, where MFe_2O_4 -based devices can be extended up to 1.8 V; (iii) enhanced chemical stability of the host electrodes; and (iv) high natural abundance, low cost and eco-friendly nature of iron [27,28]. Asymmetric supercapacitors (ASCs) combine the merits of batteries and supercapacitors to improve energy and power densities simultaneously. ASCs combine a capacitive electrode (anode) that is capable of providing fast power with a faradaic battery-type electrode (cathode) that is able to store energy to overcome the voltage limit (below 1.0 V) that limits the potential window of aqueous symmetric supercapacitors because of thermodynamic water decomposition. The wider operating voltage window greatly enhances the energy density of the device, satisfying the requirements of applications that need both fast power delivery and high storage capacity [29]. SCs are widely employed in electric and hybrid electric vehicles (EVs/HEVs). Recently, hybrid supercapacitors, also known as “SC batteries”, that combine supercapacitor and lithium-ion battery active materials within one electrode have achieved a significant improvement in energy density over traditional electrochemical capacitors. For HEV applications, the energy density of SC batteries is approaching the energy density of high-power lithium-ion batteries. Furthermore, these hybrid architectures can reduce the high self-discharge rates of EDLCs, which have high power output, long cycle life and a wide thermal operating range [30]. This review provides an extensive overview of ferrite materials and their composites for SCs, including their preparation methods, electrolyte compatibility and electrochemical properties. We discuss the structural and compositional properties of the most relevant transition metal ferrites and evaluate their performance in SC devices. In this work, we focus on the material synthesis and electrode optimization as the factors determining the overall device efficiency, highlighting the strong potential of ferrites and their composites to increase energy density, power density and long-term cycling stability.

II. FERRITE SYNTHESIS TECHNIQUES:

Ferrite nanomaterials are highly sensitive to synthesis methods, and these processes significantly affect their morphology, purity, chemical stability, and surface area. This has led to the development of numerous synthetic routes, each with distinct advantages and disadvantages for tuning material properties [31]. The synthesis routes and operational parameters are precisely tuned to control the physical and chemical properties of the ferrite nanoparticles, such as particle size distribution, morphology and phase structure. Exact material customization is possible by tuning parameters like solution pH, agitation speed, precursor concentration, dopant levels and annealing conditions [32].

III. HYDROTHERMAL/SOLVOTHERMAL METHOD

Hydrothermal and solvothermal approaches are robust wet-chemical methods for the synthesis of crystalline MFe_2O_4 NPs. They are so efficient that they can tailor particle morphology with high structural purity [33,34]. High-quality MFe_2O_4 architectures are widely prepared by hydrothermal processes because of their cost-effectiveness and



morphological versatility. Enclosed hydrothermal reactors operating above ambient thermal (greater than 100 °C) and barometric (greater than 1 atm) limits promote dissolution-recrystallization pathways in aqueous solutions in the reaction environment. Key handles such as solution pH, heating profiles, precursor ratios and vessel volume can be varied to tune the geometry of nanoparticles. Thermal annealing that follows ensures complete crystallization and produces transition metal oxide electrodes with long lifetimes for next-generation energy storage devices [35–37]. Solvothermal routes allow the precise phase and morphological control of binary spinel ferrites (XFe_2O_4 , X = Mn, Co, Ni) for supercapacitor applications. Ullah et al.[38] compared the substitution of cations and found that the Co substitution (CoFe_2O_4) showed better electrochemical activity than the Mn and Ni analogues. The as-prepared CoFe_2O_4 electrode exhibited a high specific capacitance of 1518 F/g at 0.5 A/g and showed excellent long-term cycling stability with 99.9% of the initial specific capacitance retained after 5,000 galvanostatic charge-discharge cycles. Moreover, a two-electrode symmetric supercapacitor fabricated from CoFe_2O_4 showed an energy density of 33.38 Wh/kg at a power density of 150 W/kg, thus pointing out its promise as a strong transition metal oxide platform for high-performance energy storage devices. Huang et al. [39] have synthesized 2D porous layered NiFe_2O_4 nanostructures by a simple hydrothermal route followed by calcination at 350 °C. Characterization confirmed a spinel crystal structure with a high specific surface area of 116.35 m^2/g and an interconnected mesoporous network centred at approximately 3.2 nm that readily enabled fast ion transport and electron transfer. In a three-electrode system with 6 M KOH, the NiFe_2O_4 electrode showed obvious faradaic pseudocapacitance and exhibited a high specific capacitance of 425.62 F/g at 1 A/g, a good rate capability (retention rate of 75.26% at 16 A/g) and cycling stability (retention rate of 88.61% after 3,500 cycles). Moreover, an aqueous asymmetric supercapacitor device (NiFe_2O_4 /activated carbon) was operated in a 1.6 V voltage window with a specific capacitance of 105.94 F/g at 1 A/g, a maximum energy density of 37.67 Wh/kg at a power density of 800.02 W/kg and long-term durability with 84.21% capacity retention over 4000 cycles. Zhu et al. [40] reported a facile solvothermal route to synthesize hierarchically porous CuFe_2O_4 nanospheres assembled by primary nanocrystals with an average diameter of 60 nm by using ethylene glycol, sodium acetate and polyvinylpyrrolidone (PVP) at 160 °C for 24 h. The formed 3D superstructure has a cubic spinel crystal phase, a Brunauer–Emmett–Teller (BET) specific surface area of 57.7 m^2/g and a bimodal mesoporous pore size distribution. Mechanistic studies revealed that the assembly process is achieved by basic precipitation of plate-like amorphous hydroxides, dehydration to primary CuFe_2O_4 nanocrystals and PVP-directed oriented aggregation to porous spheres; importantly, extending the reaction time beyond 24 h results in the nanospheres disassembling into approximately 10 nm aggregated nanoparticles. These materials are evaluated as electrode materials for supercapacitors in 1 M KOH. The hierarchically mesoporous network facilitates the diffusion of electrolyte into the internal active sites, leading to fast diffusion-controlled Faradaic redox reactions. The CuFe_2O_4 nanospheres showed a high specific capacitance of 289.5 F/g at a current density of 0.6 A/g, which increased to 334 F/g during the initial cycling activation (first 200 cycles) and retained 88% capacity after 600 charge–discharge cycles. This study shows that tuning nano-hierarchical architecture in inverse spinel ferrites can remarkably enhance the electrochemical charge storage performance. Pseudocapacitor electrodes of cubic spinel AB_2O_4 -type MnFe_2O_4 nanocrystallites were prepared by a simple hydrothermal route followed by post-annealing at 400 °C, as shown by Kwon et al.[41] the governing role of hydrothermal reaction temperature in the range of 140 °C to 200 °C in dictating the phase purity, morphological evolution and overall crystallinity has been elucidated. At lower reaction temperatures (≤ 160 °C), X-ray diffraction and electron microscopy have shown the presence of a secondary, electrochemically inactive Fe_2O_3 phase with rod- and plate-like structures, in addition to quasi-spherical MnFe_2O_4 particles. The synthesis temperature was increased to 180 °C and 200 °C, which completely removed the impurity phases, and pure-phase, highly crystalline MnFe_2O_4 nanostructures were produced. Transmission electron microscopy of the optimized 200 °C sample revealed uniform quasi-spherical nanoparticles with diameters ranging from 30 to 50 nm and a lattice fringe spacing of 0.256 nm, which is the characteristic (311) plane of cubic spinel manganese ferrite. The hydrothermal reaction temperature was directly correlated to the capacitive performance tested in a three-electrode configuration with an aqueous electrolyte of 2 M KOH in the potential window of 0 to 0.45 V (vs. Ag/AgCl). Galvanostatic charge-discharge measurements revealed a



stepwise increase in specific capacitance from 78.3 F/g (140 °C) and 119.8 F/g (160 °C) to 194 F/g (180 °C) at 0.5 A/g, with the highest value of 282.4 F/g for the 200 °C sample. The prominent non-rectangular redox peaks between 0.1 and 0.45 V in cyclic voltammetry indicated that fast and reversible Faradaic pseudocapacitance played a dominant role in charge storage. The optimized MnFe_2O_4 electrode delivered a stable rate capability with capacitances of 223.6, 180.4, 149.3, 128.0 and 113.3 F/g at current densities from 1.0 to 5.0 A/g, respectively. The improved pseudocapacitive behaviour of the MnFe_2O_4 nanocrystallites synthesized at 200 °C is attributed to the uniform grain size and surface reactivity, which provide a large number of electroactive sites and significantly shorten the diffusion path lengths for OH^- ions. Electrochemical impedance spectroscopy showed fast charge-transfer kinetics and efficient ion diffusion, as indicated by depressed semicircles at high frequencies and steep capacitive slopes at low frequencies. The cycling test over a long period of time at a high current density of 3 A/g showed durable stability, retaining 85.8% of the initial capacitance after 2000 continuous charge-discharge cycles with a constant coulombic efficiency of 100%. The small capacity loss after cycling was ascribed to an increase in the series cell resistance due to local mechanical degradation at the active nanomaterial/nickel foam current collector interface. Tiwari et al.[42] reported a binder-free hydrothermal synthesis strategy to directly deposit binary transition metal oxide ZnFe_2O_4 nanostructures on 3D porous nickel foam substrates, followed by post-calcination at 350 °C for 2 hours. X-ray diffraction (XRD) was used to confirm the formation of a crystalline spinel cubic phase with lattice parameter $a = b = c = 8.78\text{\AA}$. SEM analysis revealed a homogeneous coating of thin nanosheets in an interconnected and densely packed network on the nickel foam scaffold. This porous 3D architecture provides structural stability, electrolyte penetration and shortens ion diffusion pathways to maximize electroactive site availability. Electrochemical performance in a three-electrode configuration with 1 M KOH electrolyte revealed a high Faradaic pseudocapacitance contribution from reversible redox transformations of zinc and iron species. The specific capacitance of 552 F/g was obtained from CV measurements at a scan rate of 5 mV/s, while 445 F/g was obtained from GCD profiles at a current density of 1 mA/cm². The b-value of 0.72 from the power law fit ($i = av^b$) of the kinetic analysis confirmed that the charge storage is controlled by the fast surface capacitive processes instead of the diffusion-limited intercalation. In half-cell testing, the electrode showed an energy density of 72.3 Wh/kg at a power density of 0.5 kW/kg with a low equivalent series resistance ($R_s = 4.43\ \Omega$). Moreover, the long-term test showed a capacitance retention of 85% after 1000 continuous cycles at 100 mV/sec. Sethi et al.[43] synthesized the nickel ferrite (NiFe_2O_4) nanoparticles by a low-temperature, mixed-solvent (water and ethylene glycol 1:1) solvothermal method without using expensive nickel foam substrates or high electrolyte concentrations. The formation of a pure cubic inverse spinel structure with an average crystallite size of 17 nm was confirmed by X-ray diffraction (XRD) and high-resolution transmission electron microscopy (HRTEM). The field-emission scanning electron microscopy (FESEM) and TEM analysis showed the presence of homogeneously distributed spherical nanoparticles with a mesoporous structure. The nitrogen adsorption-desorption analysis showed a high Brunauer-Emmett-Teller (BET) specific surface area of 80.0 m²/g and a large pore volume of 0.54 cm³/g, which allows for efficient ion transport. These experimental observations are corroborated by Density Functional Theory (DFT) calculations, which confirm the existence of occupied electronic states close to the Fermi level and predict an increase in the quantum capacitance. Electrochemical evaluation in a three-electrode system with 2 M KOH electrolyte shows a specific capacitance of 478.0 F/g at a scan rate of 5 mV/s (from CV) and 368.0 F/g at a current density of 1 A/g (from GCD). The electrode exhibited long-term stability, maintaining 88% of its initial capacitance after 10,000 continuous charge-discharge cycles at a high current density of 8 A/g. The deconvolution of the capacitive mechanisms indicated the equal contribution of the pseudocapacitance (50.3%) and the electric double layer capacitance (49.7%). In a fabricated symmetric supercapacitor device, the material exhibited a specific capacitance of 89 F/g at 2 mV/s and 64 F/g at 0.5 A/g and 81% capacitance retention after 10,000 cycles at 8 A/g. The device configuration showed 67.9% of double-layer capacitance and 32.1% of pseudocapacitance. The combination of the low-cost solvothermal processing, high-rate capability and mechanical durability emphasizes the potential of these NiFe_2O_4 nanoparticles for practical supercapacitor applications. Yu et al.[44] prepared inverse spinel NiFe_2O_4 NPs directly on flexible carbon cloth microfibers via a CTAB-assisted hydrothermal method and thermal annealing in air. The binder-free carbon cloth



composite achieved an active material loading density of 1.55 mg/cm². Nanoparticles with diameters of 14 to 18 nm uniformly covered the three-dimensional conductive micro-architecture. The direct growth of the material on the substrate during the synthesis process eliminates the need for polymer binders or conductive additives, leading to efficient electron transport pathways, reduced internal charge-transfer resistance, and significantly improved electrolyte ion accessibility. Electrochemical evaluation in a two-electrode cell presented excellent capacitive performance in acidic (1 M H₂SO₄) and alkaline (6 M KOH) aqueous electrolytes. The electrode showed specific capacitances of 1135.5 F/g in H₂SO₄ and 922.6 F/g in KOH at a current density of 2 mA/cm². The higher storage capacity in the acidic medium is due to the smaller ionic radius of H⁺ compared to K⁺ ions that can be easily inserted, resulting in a lower equivalent series resistance of 0.25 Ω compared to 0.30 Ω in KOH. Both electrolyte systems showed high-rate capability, maintaining more than 80% of the initial capacitance (935.5 F/g in H₂SO₄ and 787.1 F/g in KOH) when the current density was increased to 100 mA/cm². Moreover, the long-term cycling tests exhibited excellent durability with the capacitance retention of 91.2% in H₂SO₄ and 91.9% in KOH after 3,000 cycles at 20 mA/cm². A symmetric all-solid-state supercapacitor consisting of two composite electrodes and a PVA-H₂SO₄ gel electrolyte separator was fabricated to evaluate its practical potential for wearable energy storage. The flexible device showed stable operation over a large 2.0 V voltage window with a volumetric capacitance of 4.09 F/cm³ and a maximum volumetric energy density of 2.07 mWh/cm³ at 2 mA/cm². It even provided an energy density of 0.56 mWh/cm³ at high operational demands of 10 mA/cm², and fully retained its electrochemical function at mechanical bending of up to 180°. Gao et al.[45] developed a binder-free structure of hierarchically mesoporous CoFe₂O₄ nanosheets grown directly on nickel foam substrates. The 2D CoFe₂O₄ nanosheets were synthesized by a surfactant-free hydrothermal reaction and post-calcination at 400 °C, with a thickness of 30–60 nm, uniform mesopores (2–4 nm pore diameter) and a BET surface area of 68.3 m²/g. This porous and interconnected network greatly reduces the diffusion path length for electrolyte ions and provides a solid structural scaffold for the fast Faradaic redox reactions of Co²⁺/Co³⁺/Co⁴⁺ and Fe³⁺/Fe⁶⁺ species. The binder-free nanosheet CoFe₂O₄ electrode in a three-electrode system with 3 M KOH electrolyte showed a specific capacitance of 503 F/g at a current density of 2 A/g. The material showed a good rate capability with the retention of 78.5% of the initial capacitance (395 F/g) even at a ten-fold increase in current density to 20 A/g. Electrochemical impedance spectroscopy (EIS) indicated low series resistance ($R_s = 0.56 \Omega$) and efficient charge transfer kinetics. To assess the practical device performance, an aqueous ASC was constructed using CoFe₂O₄ nanosheets as the positive electrode and activated carbon (AC) as the negative electrode. The CoFe₂O₄//AC ASC exhibits a maximum specific capacitance of 73.12 F/g in a wider voltage range of 0–1.5 V at a current density of 1.2 A/g. The device could reach a maximum energy density of 22.85 Wh/kg at the power density of 900 W/kg, and still have 10.67 Wh/kg at the high-power density of 6000 W/kg. Moreover, the ASC also exhibited long-term stability with 98% capacitance retention after 5,000 continuous charge-discharge cycles at 4.8 A/g. Malaie et al. [46] reported the direct growth of magnesium ferrite rose nanoflowers (MFRNs) on nickel foam (NiF) substrates via a two-step hydrothermal reaction followed by post-annealing at 400 °C. Field emission scanning electron microscopy (FESEM) revealed a micro-to-nano hierarchical architecture consisting of flower bundles (500–900 nm in diameter) built from interconnected sheet-like nanopetals with a thickness of 22–44 nm. This binder-free porous network avoided the use of polymer binders, reduced the contact resistance and facilitated fast ion diffusion. The specific capacitance of the MFRNs/NiF electrode was 240 F/g (121 mF/cm²) at a scan rate of 20 mV/sec and 250 F/g at a current density of 1 A/g in a three-electrode cell using 3 M KOH electrolyte. The electrode maintained 54% capacitance retention when the scan rate was increased ten-fold to 200 mV/s. Extended cycling over 2,000 cycles at 100 mV/s resulted in electrochemical activation rather than degradation, with capacitance increasing from 156 F/g (78 mF/cm²) to 217.7 F/g (110 mF/cm²) due to enhanced pseudocapacitive contribution and a drop in equivalent series resistance (ESR) from 2.0 to 1.7 Ω . An asymmetric supercapacitor (ASC) constructed with MFRNs/NiF as the positive electrode and activated carbon (AC) as the negative electrode operated across a voltage range of 0–1.2 V in 3 M KOH. The AC//MFRNs ASC achieved an areal capacitance of 26 mF/cm² at 0.5 mA/cm² and delivered a maximum energy density of 2.5 Wh/kg at a power density of 73.5 W/kg.



Gao et al.[47] demonstrated that tuning the reaction time at 180°C from 8 to 10 hours drives a controlled morphological evolution of nickel ferrite (NiFe_2O_4 , NFO) grown directly on graphene nanosheets (GNSs), transitioning the oxide phase from isolated nanospheres into interconnected, vertically aligned nanosheets. The capacitance-dominated behaviour of the NFO/GNSs-10 nanosheet electrode exhibited a specific capacitance of 464.15 F/g at 1 A/g, and the value was almost double that of the nano-spherical counterpart in the three-electrode testing. The improved charge storage can be ascribed to the decreased contact resistance between NFO nanosheets and GNSs and the enhanced ion diffusion coefficient. When assembled into an asymmetric supercapacitor (ASC, NFO/GNSs-10 // AC), the device delivered a maximum energy density of 14.04 Wh/kg at a power density of 70 W/kg and still retained 7.39 Wh/kg at an ultra-high-power density of 7000 W/kg. Moreover, the architecture showed excellent stability, with the specific capacitance increased to 140% of the initial value after 5,000 cycles at 0.5 A/g owing to the gradual phase activation and structure preservation. Kumar et al. [48] synthesized pure, highly crystalline mesoporous NiFe_2O_4 nanoparticles by a low-temperature one-step hydrothermal reaction at 120°C for 8 h using hexamethylenetetramine (HMT) as a structure modifier. XRD and HRTEM characterizations showed a face-centred cubic spinel structure with well-defined nanoparticles in the range of 10-15 nm in diameter. Nitrogen physisorption analysis revealed a Type IV adsorption-desorption isotherm with an H_2 hysteresis loop indicative of a loosely packed, disordered mesoporous structure. The designed structure provided a high specific surface area of 148 m^2/g , a total pore volume of 0.205 cm^3/g and a dominant pore size distribution in the range of 2–4 nm, which maximized the active surface sites and offered interconnected channels for fast electrolyte ion transport during redox cycling. The mesoporous NiFe_2O_4 electrode exhibited better electrochemical performance in the three-electrode system using 2 M KOH aqueous electrolyte. The cyclic voltammetry measurements showed distinct redox peaks, which indicated quasi-reversible Faradaic charge storage mechanisms. The galvanostatic charge–discharge measurements showed a high specific capacitance of 1040 F/g at a current density of 1 A/g, and 525 F/g was retained even at a high current load of 15 A/g. Electrochemical impedance spectroscopy highlighted rapid electron transport kinetics, showing a low internal series resistance of 0.892 Ω and a small charge-transfer resistance of 223.3 m Ω . The material kept approximately 70% capacitance after 500 charge-discharge cycles at 2 A/g, with minor degradation caused by the surface phase changes during continuous Faradaic reactions. This work in general demonstrates that the low-temperature mesoporous engineering is an effective pathway to enhance the ion diffusion kinetics and specific capacitance of pristine transition metal ferrites for supercapacitor applications.

Sr. No.	Method of Synthesis	Material	Specific Capacitance	Electrolyte	Morphology	Energy Density	Power Density	Cyclic Stability	Ref.
1	Solvothermal	MnFe_2O_4	523 F/g at 0.5 A/g	1 M KOH	Agglomerated	26.16 Wh/kg	150 W/kg	99.9% (5000 cycles)	[38]
		CoFe_2O_4	1518 F/g at 0.5 A/g	1 M KOH	Agglomerated	75.92 Wh/kg	50 W/kg	-	[38]
		NiFe_2O_4	1046 F/g at 0.5 A/g	1 M KOH	Densely packed aggregates	29.43 Wh/kg	112.5 W/kg	-	[38]
2	Hydrothermal	NiFe_2O_4	425.62 F/g at 1.0 A/g	6 M KOH	2D porous nanostructures	-	-	88.61% (3500 cycles)	[39]
3	Solvothermal	CoFe_2O_4	289.5 F/g at 0.6 A/g	1 M KOH	Porous	-	-	88% (600 cycles)	[40]
4	Hydrothermal	MnFe_2O_4	282.4 F/g at 0.5 A/g	2 M KOH	Quasi-spherical	-	-	85.8% (2000 cycles)	[41]
5	Hydrothermal	ZnFe_2O_4	552 F/g at 5 mV/s	1 M KOH	Nanosheets	72.3 Wh/kg	0.5kW/kg	85% (1000 cycles)	[42]



6	Solvothermal	NiFe ₂ O ₄	478 F/g at 5 mV/s	2 M KOH	Spherical	-	-	88% (after 10,000)	[43]
7	Hydrothermal	NiFe ₂ O ₄	1135.5 F/g	1M H ₂ SO ₄	-	-	-	91.2%	[44]
	Hydrothermal	NiFe ₂ O ₄	922.6 F/g at 2 mA	6 M KOH	-	-	-	91.9% (after 3,000 cycles)	[44]
8	Hydrothermal	CoFe ₂ O ₄	503 F/g at 2 A/g	3 M KOH	Mesoporous Nanosheets	-	-	78.5%	[45]
9	Hydrothermal	MgFe ₂ O ₄	250 F/g at 1 A/g	3 M KOH	Nanoflower	-	-	54%	[46]
10	Hydrothermal	NiFe ₂ O ₄	464.15 F/g at 1 A/g	6.0 mol L ⁻¹ KOH	Nanosheets	-	-	-	[47]
11	Hydrothermal	NiFe ₂ O ₄	1040 F/g at 1 A/g	2 M KOH	Spherical	-	-	Approximately 70% (after 500 cycles)	[48]
12	Electrodeposition	ZnFe ₂ O ₄	1093 F/g at 1 A/g	6 M KOH	Porous Nanosheet	54.6 Wh/kg	600 W/kg	93.5% (after 5,000 cycles)	[52]
13	Co-precipitation	NiFe ₂ O ₄	398 C/g at 1 A/g	6 M KOH	Porous	27.71 Wh/kg	14.49 kW/kg	Approximately 98% (after 6,500)	[55]
14	Electrodeposition	MnFe ₂ O ₄	70.1 F/g at 3.3 A/g	Alkaline electrolyte	Nanoflakes to Nanorods	35.1 Wh/kg	3.1k W/kg	81.1% (after 5,000 cycles)	[53]
15	Sol-gel	NiFe ₂ O ₄	160 F/g at 5 mV/s	-	-	5.5Wh/kg	900W/kg	-	[59]
16	sol-gel auto-combustion	CoFe ₂ O ₄	443.89 F/g at 1 A/g	-	Porous	-	-	79.35% (after 6,000 cycles)	[60]

IV. ELECTRODEPOSITION

Electrodeposition is an electrochemical process in which material is deposited on an electrode surface by the application of an external voltage. This method allows for uniform coating on even complex geometries, and precise control over the thickness and composition of the deposited layer, usually at room or moderately low temperatures [49]. The technique has attracted considerable interest because of its many advantages, including low cost, ease of operation, low energy consumption, high efficiency, and wide applicability. Another advantage of this method is that binders are not used in the preparation of the material, as binders may have negative effects on the electrochemical performance. Furthermore, the morphology of MFe₂O₄ can be easily controlled by changing parameters such as deposition time, applied potential and electrolyte concentration [50,51]. Joshi et al. [52] have developed a one-step binder-free electrodeposition method to synthesize bimetallic zinc ferrite (ZnFe₂O₄) nanosheets directly on nickel foil substrates for supercapacitor applications. Thermal annealing in air at 400 °C after the electrodeposition resulted in the formation of a pure crystalline cubic spinel phase with lattice fringe spacings of 0.28 nm and 0.24 nm corresponding to the (220) and (311) planes, respectively. The sulfate ions (SO₄²⁻) of the iron sulfate salt served as capping agents during the electrodeposition process, which inhibited the oriented crystal growth and favoured the generation of the interconnected two-dimensional nanosheet architectures. Direct growth on the nickel current collector provided seamless electrical connection without the need for polymeric binders or conductive carbon additives and without internal resistance drops during cycling. The morphology and electrochemical capability were found to be strongly dependent on the concentration of metal precursor salts in the deposition bath, keeping the Zn²⁺: Fe²⁺ molar ratio constant at 1:2. Thin and sparse distribution of nanosheets (Case 1: 633 F/g at 1 A/g) or shortened networks with irregular voids (Case 2:



883 F/g at 1 A/g) were obtained by dilute precursor solutions. In contrast, the over-concentrated solutions caused simultaneous growth, resulting in too dense and agglomerated nanosheets, which hindered the electrolyte penetration and decreased the capacity (Case 4: 673 F/g at 1 A/g). Optimized precursor concentrations of 0.075 M zinc nitrate and 0.150 M iron sulfate (Case 3) resulted in a highly interlinked, porous nanosheet network that maximized electroactive sites and shortened ion diffusion channels. Thus, the Case 3 electrode exhibited a specific capacitance of 1093 F/g at 1 A/g in a 6 M KOH electrolyte over a wide 1.2 V operating window. The optimized ZnFe_2O_4 nanosheet electrode achieved a maximum energy density of 54.6 Wh/kg at a power density of 600 W/kg and 30.8 Wh/kg even at a high-power density of 3000 W/kg in the symmetric coin cell supercapacitor test. The device also exhibited high electrochemical durability, retaining 93.5% of its initial capacitance over 5,000 charge-discharge cycles. These findings demonstrate that binder-free electrodeposition effectively resolves the low intrinsic conductivity common to transition metal ferrite slurries, establishing 2D ZnFe_2O_4 nanosheets as viable candidates for robust pseudocapacitive energy storage. Bhosale et al.[53] deposited cubic phase MnFe_2O_4 films on stainless steel (SS) substrates by electrodeposition at deposition times of 3, 4 and 5 min. The morphological characterization and XRD confirmed that the kinetics of the reaction is controlled by the deposition duration, causing the structural transition from nanoflakes to nanorods. The MnFe_2O_4 and MnO_2 active materials applied in asymmetric supercapacitors (ASCs) showed a maximum specific capacitance of 70.1 F/g at 3.3 A/g, leading to a maximum specific energy of 35.1 Wh/kg, specific power of 3.1 kW/kg, and capacitance retention as high as 81.1% over 5,000 cycles. Significantly, the conductive SS substrate did not show any redox interference in the alkaline electrolyte, and therefore all the energy storage metrics obtained were only from the active materials.

V. CO-PRECIPITATION

Through co-precipitation, spinel ferrite nanoparticles can be produced easily and affordably without compromising quality. This technique ensures high purity, structural homogeneity, and tailored particle size distribution[54]. Mordina et al. [55] reported the surfactant-assisted co-precipitation synthesis and direct growth of binder-free nickel ferrite (NiFe_2O_4) nanoparticles onto 3D porous nickel foam (Ni-foam), 2D stainless steel (SS) mesh, and nonporous SS foil substrates. The inverse spinel face-centred cubic (FCC) phase was obtained by thermal calcination at 600 °C with an average crystallite size of approximately 27.4 nm. The 3D interconnected Ni-foam/ NiFe_2O_4 positive electrode showed a high specific capacity of 398 C/g at 1 A/g current density when tested in 6 M KOH aqueous electrolyte. This performance was 2.78–3.65 times higher than that of SS mesh and SS foil electrodes because of the better interfacial bonding, lower charge-transfer resistance and easier transport of electrolyte within the 3D porous framework. An asymmetric hybrid supercapacitor device, NiFe_2O_4 // activated carbon has been fabricated to operate in a cell potential window of 0–1.43 V. The hybrid configuration yielded a maximum specific energy of 27.71 Wh/kg at 1 A/g and maximum specific power of 14.49 kW/kg at 20 A/g, and achieved excellent operational durability by maintaining approximately 98% of the initial specific capacity over 6,500 GCD cycles at a current density of 5 A/g. The charge balance between positive and negative electrodes afforded high Coulombic efficiencies of 85% at 1 A/g and 99% at ≥ 10 A/g. From an analytical point of view, this study is dedicated to the evaluation of battery-type Faradaic electrodes exclusively based on specific capacity (C/g) by integrating galvanostatic discharge curves, rather than specific capacitance (F/g), to avoid the possible capacitive overestimation, which is widely encountered in Faradaic energy storage systems.

VI. SOL-GEL

The sol-gel route is a process to synthesize materials by hydrolysis and subsequent condensation polymerization of metal alkoxide precursors. Heat treatment is applied afterwards to drive phase crystallization and purge volatile organic byproducts. The main advantages of this method are the precise stoichiometric control, the low equipment requirements, the cost-effectiveness, and the possibility to process at low temperatures (usually below 100 °C) [56]. The sol-gel technique is widely favoured for ferrite synthesis because it provides exceptional control over material



composition, phase structure, purity, and morphology. The stoichiometry and the crystallite size can be flexibly tuned by systematic variation of the sol concentration, reaction environment and annealing temperature. The synthesis is generally carried out by mixing available metal nitrate precursors with organic fuels such as urea, glycine, citric acid, hydrazine or carbohydrazide in a solvent, and adjusting pH with NH_4OH to obtain a uniform colloidal gel [57]. The sol–gel method can operate in a flexible reaction temperature range of 25–200 °C and can synthesize ferrite materials with a well-controlled morphology and uniform size distribution. These structural advantages, along with the uncomplicated nature of the process, make sol–gel a popular approach for material synthesis [58]. Soam et al. [59] synthesized a NiFe_2O_4 /graphene composite by the sol-gel method and tested its electrochemical performance in an aqueous sodium sulphate (Na_2SO_4) electrolyte. Phase purity and surface morphology were confirmed by XRD and SEM analysis, respectively. The composite showed a specific capacitance of 160 F/g at a scan rate of 5 mV/s and had the highest energy density of 5.5 Wh/kg and power density of 900 W/kg. In sol-gel synthesis, chelating agents such as citric acid coordinate cobalt and iron ions so that they form a stable sol. The solvent evaporates, and the sol condenses into a gel, ensuring a uniform distribution of the metal ions in the 3D matrix. Subsequent drying and calcination at high temperature remove residual moisture and organic matter, and promote the nucleation and crystallization of the CoFe_2O_4 phase. Tianfu Huang et al.[60] synthesized hierarchical porous CoFe_2O_4 through a citric acid-assisted sol-gel auto-combustion reaction. Thermal decomposition of ammonia and citric acid resulted in large volumes of gas to give a hierarchically porous framework. This CoFe_2O_4 structure showed excellent electrochemical performance when tested as a supercapacitor electrode. It showed a maximum specific capacitance of 443.89 F/g at 1 A/g current density, and exhibited good cycling stability with a capacity loss of only 20.65% after 6,000 cycles.

VII. SUMMERY AND OUTLOOKS

Spinel metal ferrites (MFe_2O_4 , M = Co, Ni, Mn, Zn, Mg, Cu) and their composites serve as high-performance pseudocapacitive electrodes for supercapacitors due to their multi-electron redox activity, wide operating voltage windows up to 1.8 V, chemical stability, and low cost. The crystallite size and surface morphology are directly determined by the synthetic pathways (e.g., hydrothermal/solvothermal processing, binder-free electrodeposition, co-precipitation, and sol-gel auto-combustion) and lead to high storage values such as 1518 F/g for CoFe_2O_4 and 1093 F/g for ZnFe_2O_4 nanosheets. To overcome the intrinsic conductivity limitations and the structural degradation during cycling, ferrites are composited with conductive carbon matrices (graphene, CNTs) or directly grown on 3D substrates. Future directions include the development of binder-free 3D architectures, hybridization with highly conductive matrices (MXenes, graphitic carbon) to prevent restacking of nanosheets, and standardization of performance parameters by reporting the Faradaic battery-type electrodes in specific capacity (C/g) instead of capacitance (F/g) only. The synergy between morphological engineering and direct conductive integration makes spinel ferrites scalable candidates for next-generation high-energy and high-power hybrid energy storage devices.

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