



# Electrochemical investigation of manganese ferrites prepared *via* a facile synthesis route for supercapacitor applications

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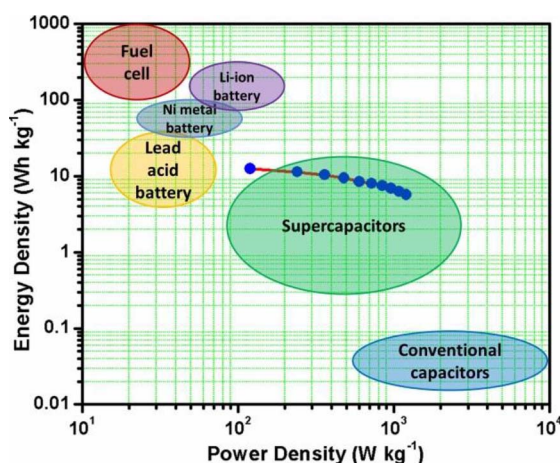
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## GRAPHICAL ABSTRACT



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## ABSTRACT

We report on a simple and facile synthesis of manganese ferrite ( $\text{MnFe}_2\text{O}_4$ ) nanoparticles by chemical co-precipitation method using 1 M NaOH as the oxidative solution. The resultant nanoparticles were characterized by using various tools like powder X-ray diffraction, Fourier transform infrared spectroscopy and scanning electron microscopy. The morphology of the resultant product was observed to be of spherical in shape with diameter of about 20–50 nm. The electrochemical performance of manganese ferrite nanoparticles was investigated by using cyclic voltammetry, charge–discharge and electrochemical impedance spectroscopy with different electrolytes, such as 1 M  $\text{LiNO}_3$ , 1 M  $\text{Li}_3\text{PO}_4$  and KOH. In a three-electrode system, a maximum specific capacitance of 173, 31 and 430  $\text{F g}^{-1}$  was attained corresponding to the electrolytes of 3.5 M KOH, 1 M  $\text{LiNO}_3$  and 1 M  $\text{Li}_3\text{PO}_4$ , respectively. Among these, 3.5 M KOH electrolyte medium exhibited excellent rate performance, evidently more than 60% of retention was observed at 10  $\text{A g}^{-1}$  due to the synergistic activities, high surface accessibility and better electronic conductivity of  $\text{MnFe}_2\text{O}_4$  nanoparticles. In addition, the fabrication of symmetric cell using  $\text{MnFe}_2\text{O}_4$  as an electrode materials with 3.5 M KOH as an electrolyte, exhibited maximum specific capacitance, high energy density and power density of 245  $\text{F g}^{-1}$ , 12.6  $\text{Wh kg}^{-1}$  and 1207  $\text{W kg}^{-1}$ , respectively. Furthermore, the specific capacitance of 105% retained after 10,000 cycles at the high current density of 1.5  $\text{A g}^{-1}$  and the coulombic efficiency of the all 10,000 cycles remains constant ( $\sim 98$ ) which clearly displayed

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the excellent electrochemical stability of  $\text{MnFe}_2\text{O}_4$  nanosphere (NS). Our results may pave the way for employing the low-cost co-precipitation method to fabricate advanced high energy storage and highly stable device with long cycle life.

## 1. Introduction

In batteries, the slow reaction kinetics due to poor conductivity restricts the overall output voltage, energy and power density of the device. The fast growing next generation needs eco-friendly, smart range of high power with maximum energy storage devices for wide range of applications such as hybrid electric vehicles, mobiles, etc. [1]. To achieve maximum energy storage capacity is a big challenge in conventional capacitors due to their low surface area. Therefore, recently many scientific communities are constantly focusing towards appropriate materials for production of high energy storing devices with maximum power delivery. In this connection, the supercapacitor is one of the appropriate contenders for this purpose. A supercapacitor is a prominent device for energy storage applications, which can overcome the bridging gap between the battery and conventional capacitors [2–4]. Supercapacitor is a promising device for energy storage applications since it exhibits attracting characteristics such as high power, minimum equivalent series resistance and extreme cycle life [5,6]. Nanomaterials play vital role in energy storage devices, especially, supercapacitors and batteries. Electrochemical supercapacitor performance is mainly controlled by various factors such as electrode material surface, morphology, current collector, separator, and electrolyte [7]. Controllably prepared nanomaterials exhibits interesting morphologies, which plays the virtual major role in the electrochemical activities [8,9]. In general, materials studied for supercapacitors are classified into two major types as follows: carbon and their derivatives, in which charge storage arises from the non-faradic reversible reaction occurring at electrode/electrolyte interface [10]. The second type, metal oxides/hydroxides/sulfides/selenides like  $\text{RuO}_2$  [11],  $\text{MnO}_2$  [12],  $\text{Co}_3\text{O}_4$  [13,14],  $\text{Ni(OH)}_2$  [15],  $\text{CoS}$  [16,17],  $\text{Ni}_3\text{S}_2$  [18], and  $\text{NiSe}_2$  [19], etc., and conductive polymers like PEDOT-PSS [20,21], polyaniline [22], polypyrrole [23,24], etc., with pseudo behavior, in which charge storage arises from the fast-reversible surface faradic reactions, occurring at electrode/electrolyte interface. The rate of redox reaction can control the electrical charge transport of pseudo materials. In supercapacitors, the surface redox reaction can take place during energy storage and hence the morphologically controlled nanomaterials were observed to have remarkable results. Cost-effective conducting polymer based electrochemical supercapacitors exhibit impressive results of both high capacitive and energy density. At the same time, they have some disadvantages such as poor cycle life and low rate performance on account of their low kinetic ion transporting nature. In general, it was observed that the metal oxides store remarkable value of capacitance, however, the high cost, lower electrical conductivity, less electrochemical stability and large scale preparation methods were limits for the device applications [25]. Therefore, recently researchers are intensively concentrating on alternative electrode materials with facile methods suitable for large scale production, low cost, earth abundant and eco-friendly in nature.

Iron is one of the most abundant elements in the earth surfaces (fourth, 5%), toxic free and low cost compare to other transition metal oxides. Iron based materials were used for a wide range of applications which comprise of medical [26], memory [27], energy and sensing devices [28,29], catalyst [30], biomedical [31], magnetic resonance imaging [32], etc. Ferrites based composite materials exhibited good electrochemical performance of both electrolytes, either organic Li-ion based battery type electrolyte [33] or aqueous electrolyte [34]. Different size and morphology of the ferrites were successfully synthesized by various methods, such as hydrothermal/solvothermal method [35], template assisted synthesis [36–38], sol-gel technique [39], co-

precipitation [32], sonochemical [40], nano-casting [30] and electrochemical synthesis [41]. All the above methods were mainly reported as the different structural formation depending upon the atmospheric conditions such as spherical, hollow spheres [38,42], sponge like [43] and nanorods/nanowires [44,45]. In supercapacitors,  $\text{RuO}_2$  is having promising characteristics; however, the unreliable nature (toxicity and cost) acts as a hindrance in further commercialization. Followed by,  $\text{MnO}_2$  which encloses promising electrochemical characteristics, thus, it is considered as an electrode material for replacement of carbon (EDLCs) and  $\text{RuO}_2$  (Pseudo) materials in supercapacitors [46]. However, as per as the commercial aspect is concerned, we should require good capacitance, high rate performance with the maximum working potential range, less toxicity with low cost. So, the various low-cost materials were taken much into consideration in electrochemical investigation. In this context, the abundant  $\text{Fe}_3\text{O}_4$  demonstrates a wide range of operating voltage in aqueous medium. Its poor rate performance and less electrochemical stability can restrict the electrode performance. In order to overcome the above issues,  $\text{Fe}^{2+}$  can be replaced with the  $\text{Mn}^{2+}$  to form the  $\text{MnFe}_2\text{O}_4$  electrode materials. Herein, these replacements of ions are of great advantage in supercapacitors owing to their combined activities of battery type iron as well as capacitor type manganese electrode evolves much higher electrochemical characteristics such as maximum energy, high power and remarkable life cycle. Evidently, Weihua et al. reported [47]  $\text{MnFe}_2\text{O}_4$ /graphene electrode materials with sphere like morphology revealed a remarkable specific capacitance  $105 \text{ F g}^{-1}$  and good electrochemical stability. Recently, Bashir et al. [48] reported  $\text{MnFe}_2\text{O}_4$  electrode materials with cluster morphology displayed a remarkable specific capacitance  $\sim 135 \text{ F g}^{-1}$  at  $5 \text{ mV s}^{-1}$  and  $\sim 25\%$  retention was observed at  $100 \text{ mV s}^{-1}$ .

In this study, we made an attempt to synthesis  $\text{MnFe}_2\text{O}_4$  nanoparticles for achieving maximum capacitance with extensive operating voltage and high rate performance in supercapacitors. We adopt a simple and scalable chemical co-precipitation method to obtain the nanoparticles. The resultant  $\text{MnFe}_2\text{O}_4$  nanospheres are characterized for their structure, morphology and their electrochemical applications. The morphology of the final product is observed to be spherical in shape and hence we labeled this material as  $\text{MnFe}_2\text{O}_4$  nanosphere (NS). Interestingly, composite of battery and supercapacitor material absorbed perfect reversible redox properties with high stability in the alkali medium.  $\text{MnFe}_2\text{O}_4$  NS is more active in some extended range of potential (1.2 V) with good rate performance and high stability compared with other metal oxides (see Table S1).

## 2. Experimental

### 2.1. Materials

$\text{MnCl}_2 \cdot 6\text{H}_2\text{O}$  (99%),  $\text{FeCl}_3 \cdot 9\text{H}_2\text{O}$  (98%),  $\text{KOH}$  (98%), 1-methyl-2-pyrrolidone (NMP) and  $\text{NaOH}$  (98%) were purchased from Merck Specialties Private Limited, India. PVDF and conductive carbon were purchased from Sigma-Aldrich. Ethanol (99.9%) was purchased from Hayman and distilled water taken into Millipore purifier purchased from Merck, India. All chemicals were of analytical grade and used without any further modification/purification.

### 2.2. Method

Chemical co-precipitation method was employed to synthesize  $\text{MnFe}_2\text{O}_4$  NS at low temperature [49]. Typically, a mixed solution of

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