



# Ultrasound-assisted synthesis of NiFe- layered double hydroxides as efficient electrode materials in supercapacitors

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## ABSTRACT

Under ultrasound irradiation, NiFe-layered double hydroxide (NiFe-LDH) nanostructures with three molar ratios and three dissimilar reaction times were prepared. The powder X-ray diffraction (XRD), field emission scanning electron microscopy (FE-SEM), and Fourier transform infrared spectroscopy (FT-IR) were employed to characterize the synthesized nanomaterials. Using a sonochemical route, various morphologies of the NiFe-LDH nanostructures without any impurity and variations in the structure were produced. During the optimization process, it was found that sonication time and reagent concentration in a fixed irradiation frequency can affect the size and the morphology of the produced nanostructures. Under ultrasound irradiation, non-aggregated particles with uniform, spherical morphology were obtained with molar ratios of 4:1 (Ni:Fe) with 45 W at 180 min. The NiFe-LDH samples were observed to be supercapacitor under a 6 M KOH solution. When morphologically-controlled NiFe-LDH samples were used, the pseudo-capacitive behavior of the nanostructures was tuned. After 3 h of ultrasonic irradiation, the optimized sample (NiFe-LDH spherical nanostructures with 4:1 M ratio) had a high value of specific capacitance ( $168 \text{ F g}^{-1}$ ).

## 1. Introduction

In recent years, the energy problem has caused considerable concern worldwide with an ever-increasing demand for high-power energy storage equipment [1]. Since supercapacitors (SCs) or electrochemical capacitors (ECs) present a greater power density and a longer life cycle compared with batteries and conventional capacitors [2–5], they have been employed in many technologies, e.g. hybrid vehicles, consumer electronics and memory backup systems [6,7]. In general, SCs are classified as electric double layer capacitors (EDLCs) or pseudocapacitors, based on their energy storage mechanism. In EDLCs, the capacitance is an outcome of charge accumulation at the electrolyte-electrode interface. Some examples of EDLC electrodes include carbon nanotubes, carbon aerogels and activated carbon. On the other hand, pseudocapacitors with the metal hydroxides, conducting polymers and metal oxides electrodes store electrochemical charge through Faradic reactions [8,9].

Moreover, the pseudo-capacitive positive electrode substances composed of nanoplate-shaped transition-metal compounds (e.g., layered double hydroxides (LDHs), and metal oxides/hydroxides) are highly promising in the field of the advanced supercapacitive materials. As a green and inexpensive electrode material in the supercapacitors, LDHs can properly utilize the transition metal atoms and show singular

structural anisotropy and remarkable specific capacitance [10,11].

Layered double hydroxides (LDHs) are layered compounds comprised of intercalated anions for charge balance and positively charged host layers [12,13]. LDHs have a general formulation of  $[\text{M}^{2+}_{1-x}\text{M}^{3+}_x(\text{OH})_2](\text{A}^{n-})_{x/n}\cdot n\text{H}_2\text{O}$ , where  $\text{A}^{n-}$  shows a charge-balancing anion,  $\text{M}^{2+}$  signifies the divalent metal cation, and  $\text{M}^{3+}$  is the trivalent metal cation. To prepare LDHs with various morphologies, numerous approaches including co-precipitation, hydrothermal, urea hydrolysis, ion exchange, and sonochemical method have already been introduced [14–16]. Since LDHs contain layered structure and demonstrate anion exchange properties, they can play the role of ion exchange hosts, fire retardant additives, drug delivery hosts, precursors of catalysts and  $\text{CO}_2$  adsorbents, and electrodes of alkaline secondary batteries [17,18].

In sonochemistry, ultrasonic power in the low frequencies under 20 kHz to 40 kHz (which are commonly used for laboratory devices) is employed [19,20]. Furthermore, the chemical reactions under sonochemical approaches are usually carried out in multi-bubble environments and are almost always accompanied by the quickly and strongly compressed and heated interior of these collapsing bubbles [21–24]. The main advantages of ultrasonication are requiring no surfactant, producing small particles and avoiding generation of high temperatures during synthesis procedures [25–27]. In addition, ultrasonication accelerates and initiates the reactions that hardly advance under normal

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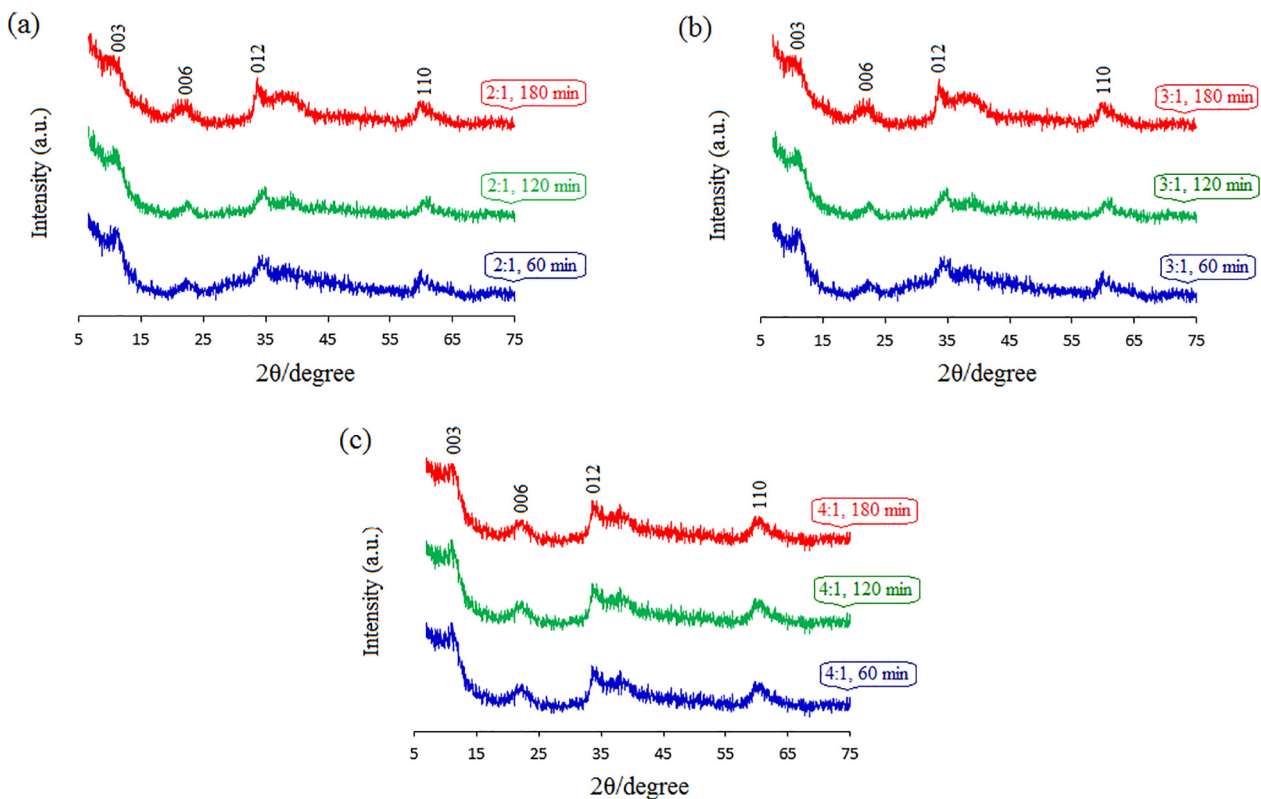


Fig. 1. XRD patterns of the NiFe-LDH in different molar ratios and times of reaction with ultrasonic illumination.

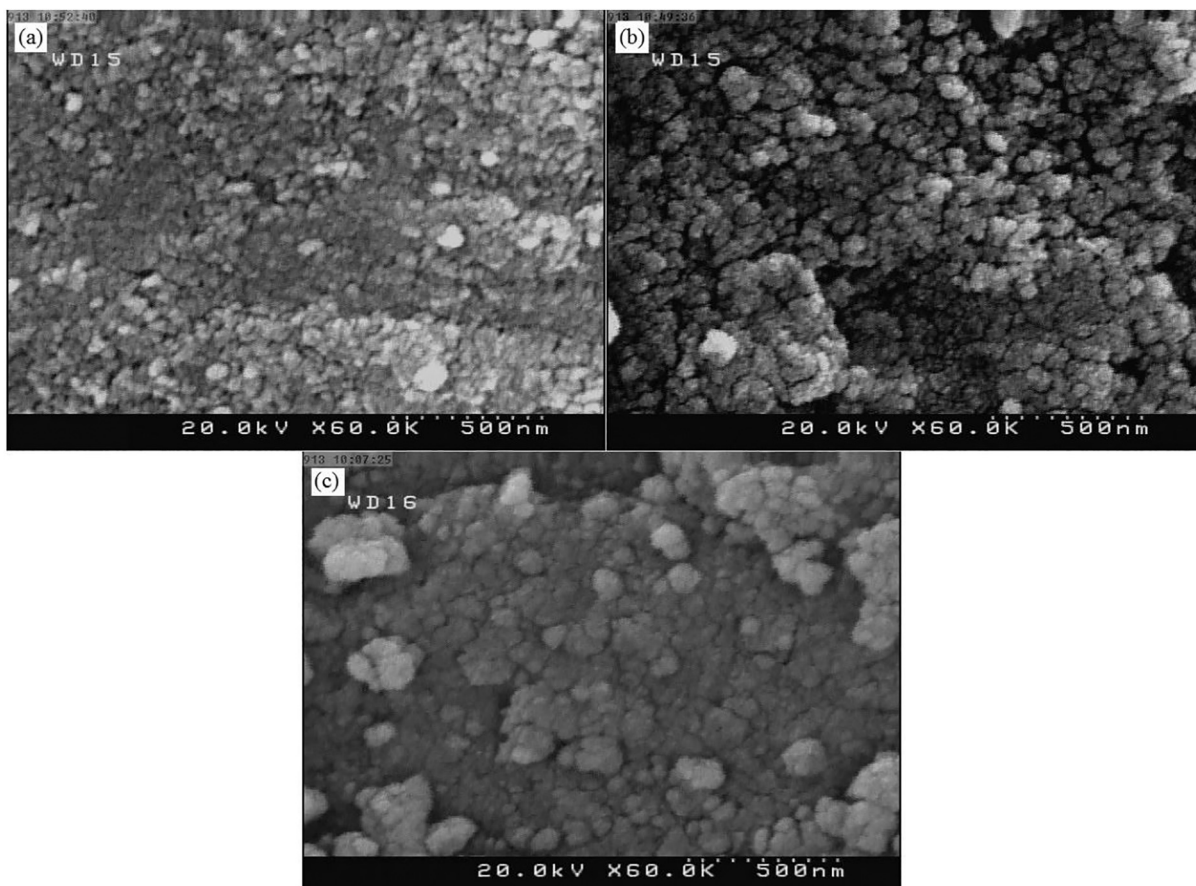


Fig. 2. FE-SEM images of NiFe-LDH with 2:1 M ratio in (a) 60 min, (b) 120 min, (c) 180 min times of reaction.

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