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CORE SHELL STRUCTURE OF NICKEL FERRITE MICROSPHERE FOR THE APPLICATION OF ENERGY STORAGE DEVICES

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Abstract

Carbon (C) coated on the surface of nickel ferrite ($NiFe_2O_4$) is a structure which could act as a protective layer to form a unique core-shell structure. The $NiFe_2O_4$ coated with C anode material shows improved lithium-storage performances since the carbon structure is considered to serve as a conductor of electrons and provide pathways for Li-ion diffusion to improve the electronic and ionic conductivities. Carbon electrodes achieve high capacitance, fast charge-discharge rate, and long cycle life for supercapacitors. In this study, pure and $NiFe_2O_4$ coated with C was synthesized by solvothermal method and followed by precipitation method. The optimum solvothermal temperatures were investigated by varying the temperatures including 110, 150 and 180 ºC respectively. The core-shell structure was prepared based on the optimum temperature. The structural properties of the final product were characterized by Fourier-transform infrared spectroscopy (FTIR), X-ray diffraction (XRD) and the surface morphology was studied by Scanning electron microscopy (SEM).

Keywords: core-shell, carbon coated, FTIR, nickel ferrite, solvothermal, SEM

Introduction

Graphite, graphene, lithium, cobalt, manganese and nickel have been focused for the energy storage applications. Nickel (Ni) is a similar chemical property of iron, cobalt, and copper. However, it is generally only stable in aqueous solution in the + 2 oxidation state. Nickel is a ferromagnetic material and it has an excellent conductor of both electricity and heat (Lebrouhi *et al.*, 2022). Mixed transition metal oxides with spinel structures such as $NiFe_2O_4$, $ZnFe_2O_4$, $CoFe_2O_4$ have high specific capacity and lower cost as compared to other classes of anode materials for battery. Generally, a spinel ferrite is described as (AB_2O_4), where A is a divalent metal ion such as Co, Ni, Zn, Mg, Cu Mn etc. Spinel ferrite possesses two interstitial sites namely tetrahedral (A) and octahedral (B) site. The spinel ferrites are classified as normal ferrite, inverse ferrite and random ferrites. Nickel ferrite possesses inverse spinel structure. It is a soft magnetic material with moderate coercivity and with low conductivity, good electrochemical stability, and abundance in nature and saturation magnetization (Bharati *et al.*, 2020; Benrabaa *et al.*, 2010).

The performance of anode materials for lithium-ion battery requires high conductivity, biocompatibility, chemical stability, corrosion resistance, and improved mechanical strength (Geofrey Sahini and Daud Lupyana, 2023). High mechanical rigidity, photostability, and thermal and electrical conductivities are characteristics of carbon-based nanomaterials. When carbon is composited with other nanoparticles, it exhibits new and improved properties such as increased electrical conductivity and structural stability (Gorgizadeh *et al.*, 2018).

Core-shell particles are made up of a shell material, such as carbon, silica, polymers, or another metal or metal oxide, around a core substance, usually a metal or metal oxide. The

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improved properties like magnetic, optical, and surface are provided by this unique core-shell structure. Therefore, the characteristics of core-shell particles are interested for solar cells, batteries, and fuel cells. When nickel ferrite with low conductivity is coated with carbon, the electrical conductivity is increased (Azizi *et al.*, 2024). In this study, $NiFe_2O_4$ is synthesized at different temperatures of solvothermal process. Depending on the optimum structural properties, it was coated with carbon for the application of energy storage device. This research is focused on the preparation and characterization of nickel ferrite coated with carbon as an anode material for the application in lithium-ion battery.

Experimental Details

Synthesis of Nickel Ferrite

Nickel ferrite was synthesized by solvothermal method (Liu *et al.*, 2020). The starting materials are nickel nitrate hexahydrate, $Ni(NO_3)_2.6H_2O$, iron nitrate nonahydrate, $Fe(NO_3)_2.9H_2O$, glycerol, C_3H_8O and isopropyl alcohol (IPA). Firstly, 0.1089 g of $Ni(NO_3)_2.6H_2O$ and 0.303 g of $Fe(NO_3)_2.9H_2O$ were added into the mixed solvent containing 24 mL of glycerol and 120 mL of isopropanol. The mixture was stirred at room temperature until to obtain the homogeneous solution. Then, solvothermal process was conducted at the reaction temperatures including 110, 150 and 180 ºC for 6 h. At the end of reaction, the yellow particles were precipitated. After that, the precipitates were centrifuged by ethanol for several times until to get the pH of the solution became neutral. The purified precipitates were dried at 60 ºC for 3 h in air and grinded by agate motor and pestle. Finally, the as-synthesized particles were calcined at 400˚C for 2 h. The reddish-brown color of nickel ferrite nanosphere was obtained. Figure 1 shows the synthesis photo of nickel ferrite microsphere.

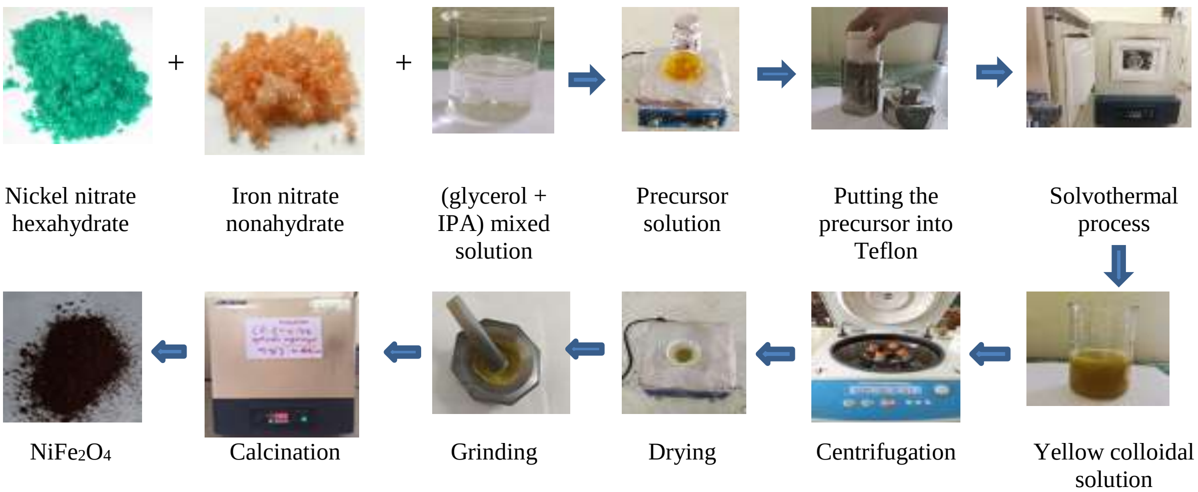


Figure 1 Synthesis photograph of nickel ferrite

Synthesis of Nickel Ferrite Coated with Carbon Microsphere

Firstly, 0.363 g of $NiFe_2O_4$ was add into the solvents containing 145.2 mL ethanol and 72.6 mL deionized water. Then, the mixed solution was sonicated for 30 min. Then, 3.63 mL of ammonium hydroxide (NH_4OH) solution was added into the previous solution and they were stirred for 15 min. After that, 0.726 g of resorcinol, $C_6H_4(OH)_2$ and 871.2 µL of formaldehyde (CH_2O) were added and continued stirring for 2 h. Next, the as-synthesized product was washed by ethanol until to obtain the pH 7. Finally, the purified product dried and annealed at 600 ºC for

2 h. The brownish black color of $NiFe_2O_4$ coated with carbon sphere was successfully obtained. The synthesis of $NiFe_2O_4$@C is presented in Figure 2.

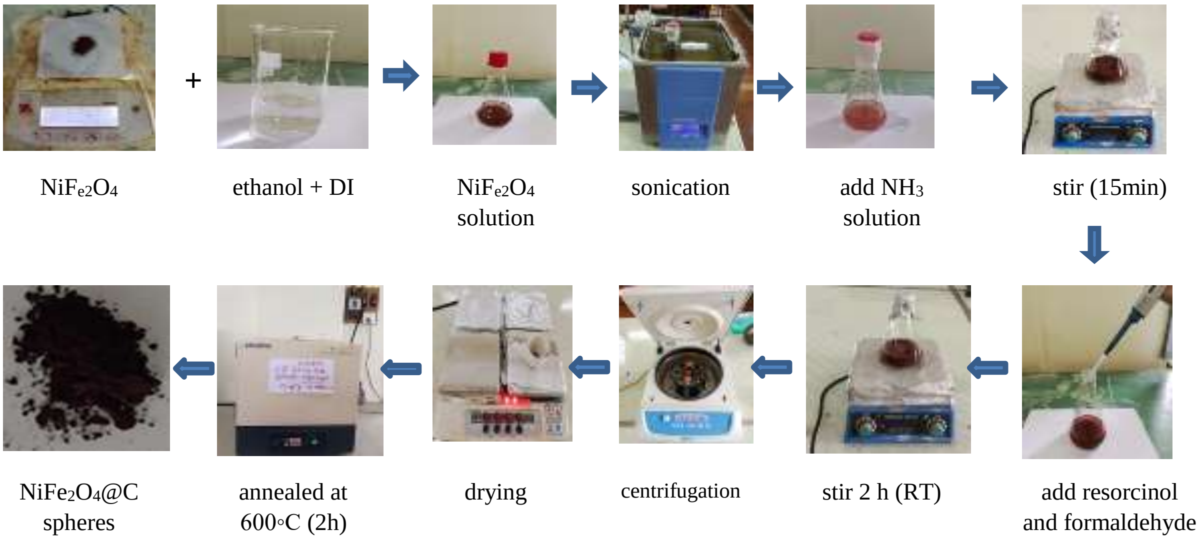
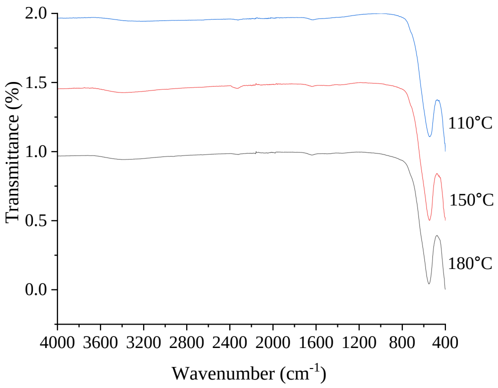


Figure 2 Synthesis photograph of nickel ferrite coated with carbon microspheres

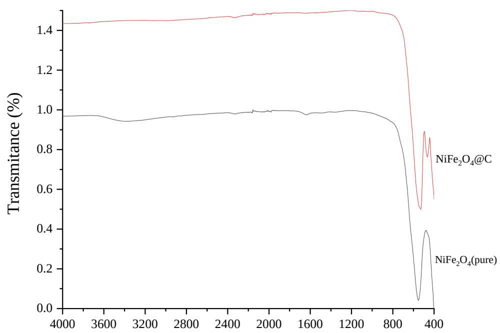
Results and Discussion

The FTIR spectra pure $NiFe_2O_4$ and $NiFe_2O_4$@C composite are illustrated in Figure 3. The peak at 554 cm^{-1} for both pure and composite samples is assigned to the bending vibrations of the metal–oxygen (MO) bond. Only one distinct peak at 554 cm^{-1} was observed in pure $NiFe_2O_4$. For composite, one more additional peak around at 470.79 cm^{-1} was occurred. These two characteristic peaks at 554 cm^{-1} and 470 cm^{-1} were corresponded to the metal-oxygen (M-O) bond stretching vibration at the tetrahedral sites and octahedral sites (Srinivasan *et al*., 2019). The difference between these absorption bands is due to the change in bond length (M-O) at the tetrahedral and octahedral site (Mounkachi *et al*., 2017). Therefore, the analysis results indicate that pure $NiFe_2O_4$ has a hematite property and the $NiFe_2O_4$@C composite has a magnetite property. These results are evidence of the color of final products of pure $NiFe_2O_4$ (reddish brown)and $NiFe_2O_4$@C (brownish black) as already mentioned the different colors in their synthesis process.



(a)

Figure 3(a) FTIR spectra of $NiFe_2O_4$ at different solvothermal reaction temperatures



(b)

Figure 3(b) FTIR transmission spectra of $NiFe_2O_4$ and $NiFe_2O_4$@C

XRD is used to examine the crystal structure, phase formation and additionally confirmation of FTIR observation. Figure 4 shows XRD spectra of $NiFe_2O_4$ and $NiFe_2O_4$@C composite. The X-ray diffraction angles of 30.30, 35.69, 43.38, 57.38 and 63.02 degrees were related to (220), (311), (400), (511) and (440) planes. These Miller indices were informed that the formation of cubic structure of pure and composite of nickel ferrite (Mohan *et al*., 2019). The most dominant peaks were occurred at (311) plane. Among the different reaction temperatures, the better crystallinity of spinel ferrite structure was observed at 180 °C. Therefore, reaction temperature 180 ºC was the optimum temperature. The peaks and relative intensities obtained for $NiFe_2O_4$ and $NiFe_2O_4$@C composite were well-matched with the standard library file of ICDD PDF databases number-5910064. The XRD spectrum of $NiFe_2O_4$@C composite was almost as the same as pure nickel ferrite except the diffraction peak in (222) plane at 37.33 degree. This plane was the evidence of carbon peak which is well agreed with FTIR results. The compared XRD spectra of pure and coated with carbon is presented in Figure 5. The crystallite size of the materials is estimated by using the Debye Scherrer Equation (Bokuniaeva and Vorokh, 2019).

$$D = \frac{K\lambda}{\beta \cos\theta} \tag{1}$$

Where,

λ = the wavelength of the incident X-ray beam (1.5405 Å) for $CuK_{\alpha 1}$

K = shape factor

β = FWHM (full width at half maximum) (radian)

θ = diffraction angle (radian)

D = crystallite size (nm)

Average crystallite sizes of nickel ferrite at different reaction temperature is listed in Table 1. The smallest crystallite size was obtained at 180 ºC. Therefore, pure nickel ferrite synthesized at 180 ºC was chosen to composite with carbon. For the composite, the crystallite size was obtained about 26.4 nm.

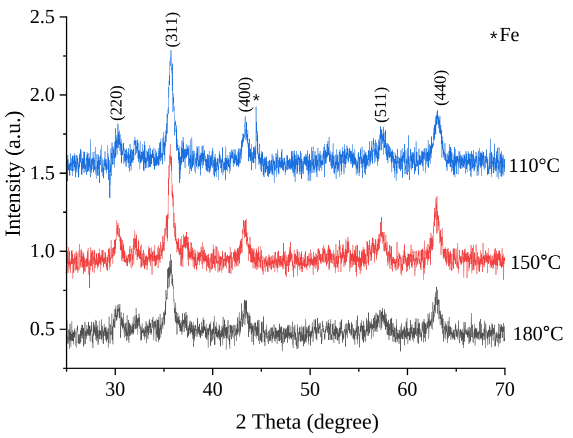


Figure 4 XRD spectra of $NiFe_2O_4$ at different solvothermal reaction temperatures

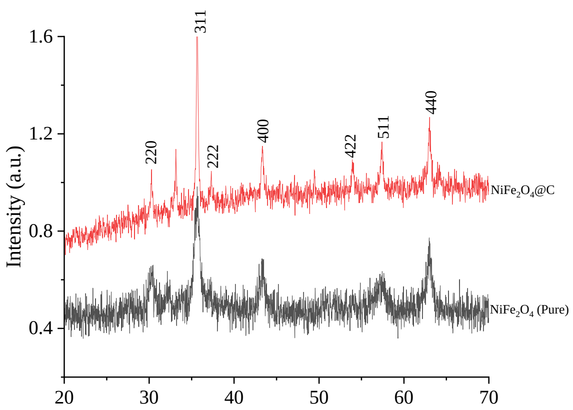
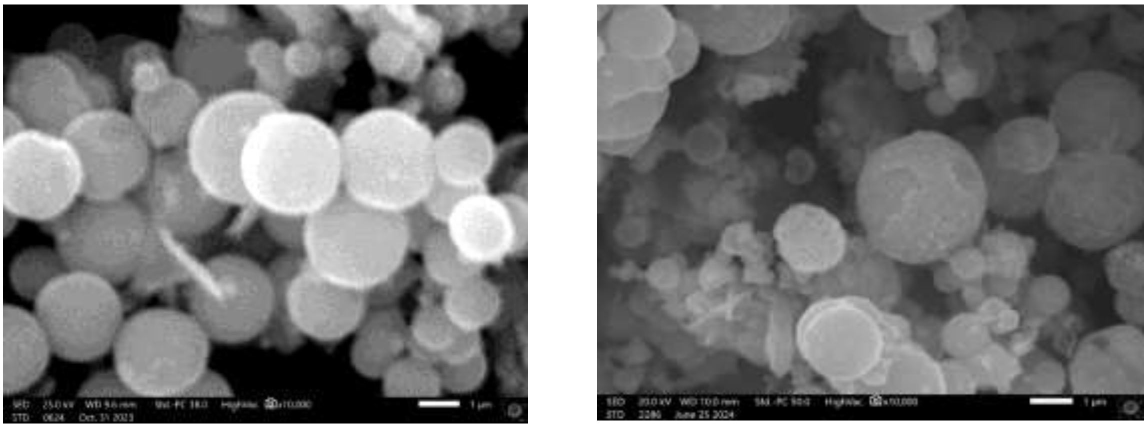


Figure 5 XRD spectra of $NiFe_2O_4$ and $NiFe_2O_4$@C

Table 1. Average crystallite size of nickel ferrite at different reaction temperatures

Solvothermal temperature (ºC)	Average crystallite size (nm)
110	12.2
150	10.1
180	8.59

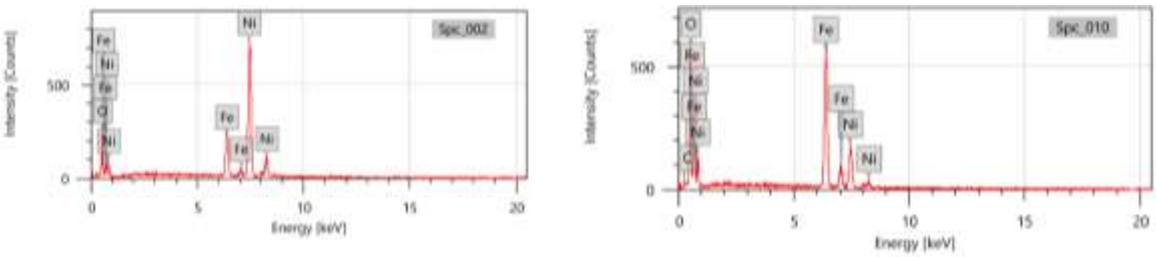
Scanning electron microscopy–energy dispersive X-ray spectroscopy (*SEM-EDX*) analysis is performed in order to investigate the surface morphologies and qualitative elemental analysis of the materials. According to the SEM image, both pure and composite are found out to be spherical shape. SEM image of $NiFe_2O_4$ calcined at 400 ºC for 2 h and $NiFe_2O_4$@C composite at 600 ºC for 2 h are display in Figure 6. The average diameter of grain size of pure $NiFe_2O_4$ was 1768 nm and $NiFe_2O_4$@C composite was 1899 nm, respectively. In the surface of composites, the spheres are relatively rough because NiFe-glycerate nanospheres encountered a hydrolysis reaction in order form the form hydroxide sheets in the solvent (Liu *et al.*, 2020). Thus, the diameters of nickel ferrite coated with carbon spheres were substantially large. Besides, the surfaces were also relatively rough, indicating that the carbon layer was uniformly covered to the surface of nickel ferrite. As the result, the core-shell structure of $NiFe_2O_4$@C was successfully obtained.



(a) (b)

Figure 6. SEM image of (a) NiFe2O4 and (b) NiFe2O4@C

Figure 7 presents the EDX spectrum of pure and composite materials. Only the particular elements were composed in their materials. These results were additionally supported to the previous observation of XRD and FTIR results. Therefore, the final product of each synthesis could be confirmed that they were high purity materials.



(a) (b)

Figure 7 EDX spectrum of (a) $NiFe_2O_4$ and (b) $NiFe_2O_4$@C

Conclusions

Nickel ferrite ($NiFe_2O_4$) spheres was synthesized by solvothermal method at different reaction temperatures including 110, 150 and 180 ºC. Depending on the optimum temperature, carbon coating process is performed onto the $NiFe_2O_4$. The structural properties of $NiFe_2O_4$ and $NiFe_2O_4$@C composites are characterized by FTIR, XRD and SEM. According to XRD result, the cubic and inverse spinel structure of composite materials was successfully obtained. Debye Scherrer formula suggested that the crystallite of $NiFe_2O_4$ was the smallest at the reaction temperature 180 ºC. FTIR result additionally evidenced that the functional group of targeted compounds and properties of pure $NiFe_2O_4$ is the hematite with paramagnetic core and $NiFe_2O_4$@C is the magnetite with ferromagnetic core-shell materials respectively. SEM image clearly confirmed that the core-shell structure $NiFe_2O_4$@C. EDX spectrum showed the elements composed in pure and composite materials. Therefore, all characterization results are directed to the formation of core shell materials which can be utilized the anode materials in the lithium ion battery.

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